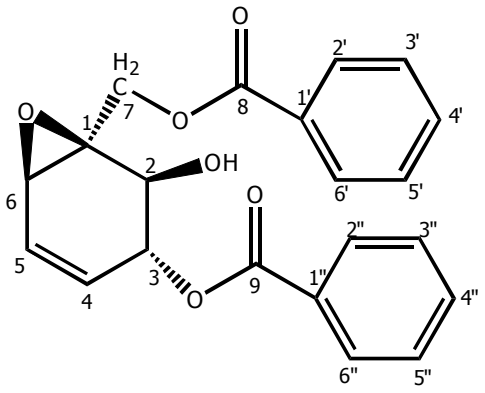
CHAPTER 3

RESULTS AND DISCUSSION

3.1 Structural elucidation of compounds from the leaves of U. purpurea Blume

The white solid precipitate was purified by PLC to yield two compounds, **SAH1** and **SAH2**. The hexane extract of leaves of *U. purpurea* Blume was subjected to column chromatography and PLC to give one compound , **SAH3**. The methylene chloride extract of leaves of *U. purpurea* Blume was subjected to column chromatography and/or crystallization to give one compound, **SAC1**. The methanol extract of leaves of *U. purpurea* Blume was subjected to column chromatography and/or crystallization to give one compound, **SAM1**. Their structures were determined using 1D and 2D NMR spectroscopic data.

3.1.1 Compound SAH1



Compound **SAH1** was obtained as a white solid, mp. = 149-150 ^{o}C, $[\alpha]_D^{25.3}$ = +51.99^{o} (*c* = 0.327, $CHCl_3$). The molecular formula was determined as $C_{21}H_{18}O_6$ by EIMS ($[M^{+}+1]$ *m/z* 367). The IR spectrum (Fig.4) indicated hydroxyl group (3441 cm^{-1}), an ester group (1728, 1723 and 1279 cm^{-1}), and a monosubstituted phenyl ring (1682 and 711 cm^{-1}). The presence of ester carbonyl carbon at δ166. 87 and 166.20 from ^{13}C NMR spectrum supported the above conclusion. The UV spectrum (Fig. 3) showed benzoyl chromophore maxima at 273, 228 and 202 nm.

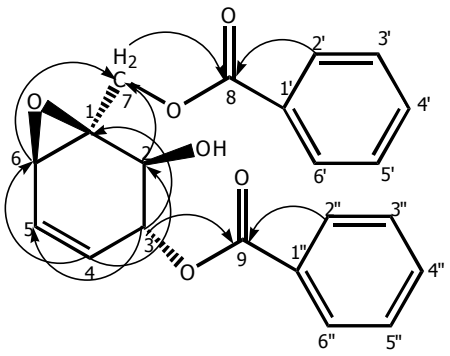
The ^{13}C NMR spectrum (see Table 2, Fig. 6) showed 17 signals for 21 carbons. Analysis of the DEPT-90^{o} and DEPT-135^{o} (see Table 2, Fig. 7) spectra of this compound suggested the presence of one methylene carbon (δ 62.91), eleven signals for fifteen methine carbons (δ 13 3 .44, 133.38, 132.96, 129.84 (2xC), 129.78 (2xC), 128.47 (2xC), 128.43 (2xC), 124.73, 74.82, 71.06 and 54.20) and five quaternary carbons (δ 166.87, 166.20, 129.44, 129.40 and 59.49).

The ^{1}H NMR spectrum (see Table 5, Fig. 5) of **SAH1** showed the presence of one hydroxyl (δ 3 .17, 1H, *d*, *J* = 6 Hz) and ten signals of aromatic protons of two monosubstituted phenyl rings (δ 7. 4 5, 4H, *m*), (δ 7.58 , 2H, *m*) and (δ 8.06, 4H, *m*). Two olefinic protons on the cyclohexene ring appeared at δ 5.91 (1H, *dt*, *J* = 10,

2 Hz) and 6.10 (1H, *ddd*, *J* = 10, 4, 2 Hz) which were assigned to H-4 and H-5, respectively.

The coupling constant between H-4 and H-5 was 10 Hz indicating that these protons were *cis*-protons. The epoxy proton resonated at δ3.60 (1H-6, *dd*, *J* = 4, 2 Hz). The oxymethine protons at C-2 and C-3 resonated at δ4.33 (1H, *dd*, *J* = 8, 6 Hz) and 5.67 (1H, *ddd*, *J* = 8, 3, 2 Hz), respectively. The proton at C-3 (δ5.67, 1H, *ddd*, *J* = 8, 3, 2 Hz) appeared at the lower field than H-2 (δ4.33, 1H, *dd*, *J* = 8, 6 Hz) suggesting that C-3 carried the electron withdrawing group, such as benzoyl group.

The complete assignment of ^{13}C and ^{1}H NMR (see Table 5, Fig. 5 and 6) signals were made with the information from ^{1}H-^{1}H COSY (see Table 3, Fig. 8), HMQC (Fig. 9) and HMBC spectrum (see Table 4, Fig. 10). In the HMBC spectrum the carbon signals at δ59.49 (C-1), 71.06 (C-2), 132.96 (C-4), 124.73 (C-5) and 166.87 (C-9) showed the correlation peaks with the H-3 (δ5.67), indicating that the benzoyl group was attached to the C-3 (74.82). The carbon signals at δ59.49 (C-1), 71.06 (C-2), 54.20 (C-6) and 166.20 (C-8) showed the correlation peaks with the methylene proton, H-7a (4.48) and H-7b (5.00), confirming that this methylene proton was attached to the C-1 (59.49) of cyclohexene oxide ring and C-8 (166.20) of the benzoyl group. From NOE experiment (Fig. 11 and 12), irradiation of H-7b (δ 5.00) showed enhancement of H-7a (δ4.48) and H-2 (δ4.33). Irradiation of H-7a (δ 4.48) showed enhancement of H-7b (δ5.00) and H-6 (δ3.60). Irradiation of H-6 (δ 3.60) showed enhancement of H-7a (δ4.48) and H-5 (δ6.10). Irradiation of H-3 (δ 5.67) showed enhancement of H-4 (δ5.91). Irradiation of H-2 (δ4.33) showed enhancement of H-7b (δ5.00). Thus, both substituents; benzoyloxymethyl and benzoyl groups at C-1 (59.49) and C-3 (74.82) are on the same side.



Selected HMBC Correlation

Comparison of ^{1}H NMR spectral data between compound **SAH1** and **(+)-pipoxide** (Joshi *et al*., 1979) (see Table 6), showed similarity. The relative stereochemistry of compound **SAH1** was deduced with optical rotation $[\alpha]_D^{25.3}$ = +51.99^{o} (*c* = 0.327, $CHCl_3$), this being almost identical to the reported value, $[\alpha]_D^{23}$ = +53^{o} (*c* = 0.02, $CHCl_3$) (Holbert *et al*., 1979). Thus compound **SAH1** was identified as **(+)-pipoxide.**

Table 2 ^{13}C and DEPT spectral data of compound **SAH1**

δ_C	**DEPT-90^{o}**	**DEPT-135^{o}**	**Type of Carbon**
166.87			C
166.20			C
133.44	133.44	133.44	CH
133.38	133.38	133.38	CH
132.96	132.96	132.96	CH
129.84	129.84	129.84	CH
129.78	129.78	129.78	CH
129.44			C
129.40			C
129.40			C

Table 2 (Continued)

δ_C	**DEPT-90°**	**DEPT-135°**	**Type of Carbon**
128.47			CH
128.43	128.47	128.47	CH
124.73	128.43	128.43	CH
74.82	124.73	124.73	CH
71.06	74.82	74.82	CH
62.91	71.06	71.06	CH_2
59.49		62.91	C
54.20	54.20	54.20	CH

Table 3 500 MHz COSY correlation of some protons of compound **SAH1**

δ_H **(ppm)**	**Proton correlation with** δ_H **(ppm)**
H-2 (4.33)	H-3 (5.67), 2-OH (3.17)
H-3 (5.67)	H-2 (4.33), H-4 (5.91), H-5 (6.10)
H-4 (5.91)	H-3 (5.67), H-5 (6.10), H-6 (3.60)
H-5 (6.10)	H-3 (5.67), H-4 (5.91), H-6 (3.60)
H-6 (3.60)	H-4 (5.91), H-5 (6.10)
H-7a (4.48)	H-7b (5.00)
2-OH (3.17)	H-2 (4.33)
H-2', H-6' (8.06)	H-3', H-5' (7.45), H-4' (7.58)
H-2", H-6" (8.06)	H-3", H5" (7.45), , H-4" (7.58)
H-3', H-5' (7.45)	H-2', H-6' (8.06), H-4' (7.58)
H-3", H5" (7.45)	H-2", H-6" (8.06), H-4" (7.58)
H-4' (7.58)	H-2', H-6' (8.06), H-3', H-5' (7.45)
H-4" (7.58)	H-2", H-6" (8.06), H-3", H5" (7.45)

Table 4 Major HMBC correlation of compound **SAH1**

Position	δ_H (ppm)	δ_C (ppm)
1	-	-
2	4.33 (1H, *dd*, *J* = 8, 6 Hz)	C-3 (74.82) and C-7 (62.91)
3	5.67 (1H, *ddd*, *J* = 8, 3, 2 Hz)	C-1 (59.49), C-2 (71.06), C-4 (132.96), C-5 (124.73) and C-9 (166.87)
4	5.91 (1H, *dt*, *J* = 10, 2 Hz)	C-2 (71.06), C-5 (124.73) and C-6 (54.20)
5	6.10 (1H, *ddd*, *J* = 10, 4, 2 Hz)	C-3 (74.82), C-4 (132.96) and C-6 (54.20)
6	3.60 (1H, *dd*, *J* = 4, 2 Hz)	C-1 (59.49), C-4 (132.96), C-5 (124.73) and C-7 (62.91)
7	4.48 (1H, *d* (AB), *J* = 12 Hz) , 5.00 (1H, *d* (AB), *J* = 12 Hz)	C-1 (59.49), C-2 (71.06), C-6 (54.20) and C-8 (166.20)
8	-	-
9	-	-
1'	-	-
2', 6'	8.06 (2H, *m*)	C-8 (166.20)
3', 5'	7.45 (2H, *m*)	-
4'	7.58 (1H, *m*)	-
1"	-	-
2", 6"	8.06 (2H, *m*)	C-9 (166.87)
3", 5"	7.45 (2H, *m*)	-
4"	7.58 (1H, *m*)	-
2-OH	3.17 (1H, *d*, *J* = 6 Hz)	C-1 (59.49), C-2 (71.06) and C-3 (74.82)

Table 5 ^{1}H and ^{13}C NMR spectral data of compound **SAH1**

Position	δ_C [#] (ppm)		δ_H (ppm)
1	59.49	C	-
2	71.06	CH	4.33 (1H, *dd*, *J* = 8, 6 Hz)
3	74.82	CH	5.67 (1H, *ddd*, *J* = 8, 3, 2 Hz)
4	132.96	CH	5.91 (1H, *dt*, *J* = 10, 2 Hz)
5	124.73	CH	6.10 (1H, *ddd*, *J* = 10, 4, 2 Hz)
6	54.20	CH	3.60 (1H, *dd*, *J* = 4, 2 Hz)
7	62.91	CH_2	4.48(1H, *d* (AB), *J* = 12 Hz)
			5.00 (1H, *d* (AB), *J* = 12 Hz)
8	166.20	C	-
9	166.87	C	-
1'	129.40	C	-
2', 6' [a]	129.78	CH	8.06 (2H, *m*)
3', 5' [b]	128.43	CH	7.45 (2H, *m*)
4' [c]	133.38	CH	7.58 (1H, *m*)
1"	129.44	C	-
2", 6" [a]	129.84	CH	8.06 (2H, *m*)
3", 5" [b]	128.47	CH	7.45 (2H, *m*)
4" [c]	133.44	CH	7.58 (1H, *m*)
2-OH	-	-	3.17 (1H, *d*, *J* = 6 Hz)

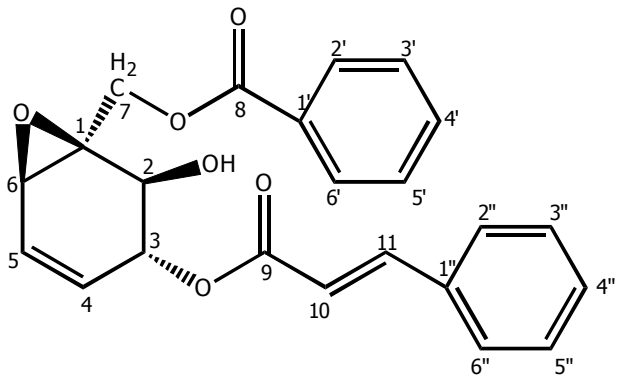
[#] Carbon type deduced from DEPT experiment.

[a, b, c] May be interchangeable.

Table 6 Comparison of ^{1}H NMR spectral data between compound **SAH1** and **(+)-pipoxide**

Position	Compound SAH1, δ_H (ppm)	Pipoxide, δ_H (ppm)
1	-	-
2	4.33 (1H, *dd*, *J* = 8, 6 Hz)	4.32 (1H, *dd*, *J* = 8.0, 6.0 Hz)
3	5.67 (1H, *ddd*, *J* = 8, 3, 2 Hz)	5.68 (1H, *dt*, *J* = 8.0, 2.5, 2.0 Hz)
4	5.91 (1H, *dt*, *J* = 10, 2 Hz)	5.90 (1H, *dt*, *J* = 10.0, 2.0, 1.75 Hz)
5	6.10 (1H, *ddd*, *J* = 10, 4, 2 Hz)	6.10 (1H, *ddd*, *J* = 10.0, 3.75, 2.5 Hz)
6	3.60 (1H, *dd*, *J* = 4, 2 Hz)	3.60 (1H, *dd*, *J* = 3.75, 1.75 Hz)
7	4.48 (1H, *d* (AB), *J* = 12 Hz)	4.48 (1H, *d* (AB), *J* = 12.0 Hz)
	5.00 (1H, *d* (AB), *J* = 12 Hz)	5.10 (1H, *d* (AB), *J* = 12.0 Hz)
8	-	-
9	-	-
1' / 1"	-	-
2', 6' / 2", 6"	8.06 (4H, *m*)	
3', 5' / 3", 5"	7.45 (4H, *m*)	7.3-8.1 (10 H, *m*)
4' / 4"	7.58 (2H, *m*)	
2-OH	3.17 (1H, *d*, *J* = 6 Hz)	3.24 (1H, *d*, *J* = 6.0 Hz)

3.1.2 Compound SAH 2



Compound **SAH2** was obtained as a white solid, mp : 132-134°C, $[\alpha]_D^{27.3}$ = +50° (*c* = 0.04, $CHCl_3$). The high resolution mass spectrum of this compound (Fig. 24) showed the molecular ion peak at 393.1338 *m/z* [M^++1], thus this compound had molecular formula $C_{23}H_{20}O_6$. The IR spectrum (Fig. 15) showed absorption bands at 3839 cm^{-1} (broad OH stretching), 1716 cm^{-1} (C=O stretching), 1273 (C-O stretching), 1631 and 711 cm^{-1} (C=C stretching and C-H bending) corresponding to a hydroxyl group, a carbonyl group of ester and a monosubstituted phenyl ring, respectively. The presence of ester carbonyl carbons at δ166 .20 and 167.32 from ^{13}C NMR spectrum supported the above conclusion. The UV spectrum (Fig. 14) showed maxima at 276, 235, 222 and 205 nm.

The ^{13}C NMR spectrum (see Table 7, Fig. 17) showed 19 signals for 23 carbons. Analysis of the DEPT–90° and DEPT–135° spectra (see Table 7, Fig. 18) of this compound suggested the presence of one methylene carbon (δ 62.92), thirteen signals for seventeen methine carbons (δ 146 .31, 133.43, 133.12, 130.66, 129.83 (2xC), 128.97 (2xC), 128.51 (2xC), 128.25 (2xC), 124.64, 117.21, 74.40, 71.10 and 54.25) and five quaternary carbons (δ16 7 .32, 166.20, 134.11, 129.51 and 59.55).

The ^{1}H NMR spectrum (see Table 10, Fig. 16) recorded in $CDCl_3$ was a typical of cyclohexene oxide. Four olefinic protons appeared at δ 5.86 (1H, *dt*, *J* = 9.9, 1.8 Hz), 6.08 (1H, *ddd*, *J* = 9.9, 3.6, 2.7 Hz), 6.47 (1H, *d*, *J* = 15.9 Hz) and 7.76 (1H, *d*, *J* = 15.9 Hz) which were assigned to H-4, H-5, H-10 and H-11, respectively. The coupling constant between H-4 and H-5 was 9.9 Hz indicating that these protons were *cis*-protons and the coupling constant between H-10 and H-11 was 15.9 Hz suggesting that these protons were *trans*-protons. The chemical shift of H-10 and H-11 appeared at the lower field than H-4 and H-5 indicating that H-10 and H-11 were attached to the electron withdrawing group , such as carbonyl group and phenyl group. The epoxy methine proton and two oxymethine protons appeared at δ 3.59 (1H-6, *dd*, *J* = 3.9, 1.8 Hz), 4.25 (1H-2, *d*, *J* = 8.1 Hz) and 5.56 (1H-3, *dt*, *J* = 8.1, 2.1 Hz) , respectively. Prochiral methylene proton, AB system, resonated at δ 4.48 (1H, *d*, *J* = 12 Hz) and 5.00 (1H, *d*, *J* = 12 Hz). The ten methine aromatic protons appeared at δ 8.06 (2H, *m*), 7.46 (2H, *m*), 7.59 (1H, *m*), 7.53 (2H, *m*), 7.40 (2H, *m*) and 7.39 (1H, *m*) indicating that there were two monosubstituted benzene.

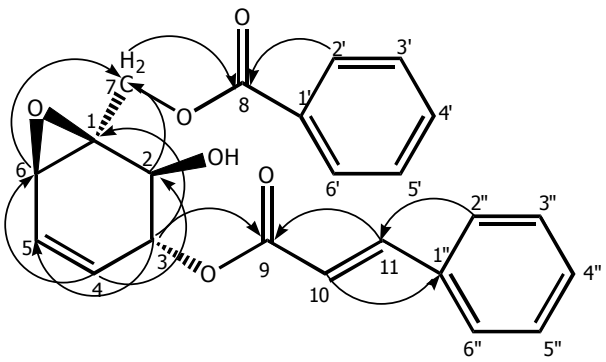
The complete assignment of ^{13}C and ^{1}H NMR (see Table 10, Fig. 16 and 17) signals were made with the information from ^{1}H-^{1}H COSY (see Table 8, Fig.19), HMQC (Fig. 20) and HMBC spectrum (see Table 9, Fig. 21). In the HMBC spectrum the carbon signals at δ 59.55 (C-1), 71.10 (C-2), 54.25 (C-6) and 166.20 (C-8) showed the correlation peaks with H-7a (4.48) and H-7b (5.00), indicating that the benzoyloxymethyl group was attached to the C-1 (59.55) of the cyclohexene oxide ring. The carbon signals at δ 59.55 (C-1), 71.10 (C-2), 133.12 (C-4), 124.64 (C-5) and 167.32 (C-9) showed the correlation peaks with the H-3 (5.56), confirming that the *trans*-cinnamoyl group was attached to the C-3 (74.40) of the cyclohexene oxide ring.

From NOE experiment (Fig.22 and 23), irradiation of H-7a (δ 4.48) showed enhancement of H-7b (δ 5.00) and H-6 (δ 3.59). Irradiation of H-7b (δ 5.00)

showed enhancement of H-7a (δ 4.48). Irradiation of H-6 (δ 3.59) showed enhancement of H-7a (δ4.48) and H-5 (δ 6.08). Irradiation of H-3 (δ 5.56) showed enhancement of H-4 (δ 5.86). Irradiation of H-2 (δ 4.25) showed enhancement of H-7b (δ 5.00). This result implied that the substituents at C-1 (59.55) and at C-3 (74.40) are on the same side (α-oriented) and the relative stereochemistry of this compound should be the same as **SAH1**.

Compound **SAH2**, a derivative of compound **SAH1**, showed similar characteristic bands in IR and UV spectrum with those of **SAH1**. Comparison of the ^{1}H NMR spectral data (see Table 11) of the two compounds revealed close structural similarity. Difference in the spectrum of compound **SAH2** was shown as additional signals of *trans*-olefinic methine protons at δ 6.47 (1H , *d*, *J* = 15.9 Hz) and 7.76 (1H, *d*, *J* = 15.9 Hz) attached to carbon at δ 117.21 and 146.31, respectively, which were not observed in compound **SAH1**.

The HMBC correlation of compound **SAH2** showed the same correlation with compound **SAH1** except additional correlations of H-10 and H-11. Correlation of H-10 (δ 6.47) with C-1" (δ 134.11) of phenyl ring, C-11 (δ 146.31) and C-9 (δ 167.32, a carbonyl carbon); of H-11 (δ 7.76) with C-9 (δ 167.32) and C-10 (δ 117.21) confirmed the position of olefinic protons. Compound **SAH2** has not been reported before. It would be designated as "Cinnamoxide".This compound supported a biosynthetic pathway in **scheme 4** (Cole, *et al.*, 1981).



Selected HMBC Correlation

Table 7 ^{13}C and DEPT spectral data of compound **SAH2**

δ_C	**DEPT-90°**	**DEPT-135°**	**Type of carbon**
167.32			C
166.20			C
146.31	146.30	146.31	CH
134.11			C
133.43	133.42	133.43	CH
133.12	133.10	133.11	CH
130.66	130.65	130.66	CH
129.83	129.81	129.82	CH
129.51			C
128.97	128.96	128.96	CH
128.51	128.50	128.50	CH
128.25	128.23	128.24	CH
124.64	124.62	124.62	CH
117.21	117.16	117.17	CH
74.40	74.36	74.37	CH
71.10	71.07	71.07	CH
62.92		62.89	CH_2
59.55			C
54.25	54.24	54.24	CH

Table 8 300 MHz COSY correlation of some protons of compound **SAH2**

δ_H (ppm)	Proton Correlation with δ_H (ppm)
H-2 (4.25)	H-3 (5.56)
H-3 (5.56)	H-2 (4.25), H-4 (5.86), H-5 (6.08)
H-4 (5.86)	H-3 (5.56), H-5 (6.08), H-6 (3.59)
H-5 (6.08)	H-3 (5.56), H-4 (5.86), H-6 (3.59)
H-6 (3.59)	H-4 (5.86), H-5 (6.08)
H-7a (4.48)	H-7b (5.00)
H-10 (6.47)	H-11 (7.76)
H-2', H-6' (8.06)	H-3', H-5' (7.46)
H-2", H-6" (7.53)	H-3", H-5" (7.40)

Table 9 Major HMBC correlation of compound **SAH2**

Position	δ_H (ppm)	δ_C (ppm)
1	-	-
2	4.25 (1H, *d*, *J* = 8.1 Hz)	C-3 (74.40), C-7 (62.92)
3	5.56 (1H, *dt*, *J* = 8.1, 2.1 Hz)	C-1 (59.55), C-2 (71.10), C-4 (133.12), C-5 (124.64) and C-9 (167.32)
4	5.86 (1H, *dt*, *J* = 9.9, 1.8 Hz)	C-2 (71.10), C-5 (124.64) and C-6 (54.24)
5	6.08 (1H, *ddd*, *J* = 9.9, 3.6, 2.7 Hz)	-
6	3.59 (1H, *dd*, *J* = 3.9, 1.8 Hz)	C-1 (59.55), C-4 (133.12), C-5

		(124.64) and C-7 (62.92)

Table 9 (Continued)

Position	**δ_H (ppm)**	**δ_C (ppm)**
7	4.48 (1H, *d* (AB), *J* = 12 Hz)	C-1 (59.55), C-2 (71.10), C-6
	5.00 (1H, *d* (AB), *J* = 12 Hz)	(54.25) and C-8 (166.20)
8	-	-
9	-	-
10	6.47 (1H, *d*, *J* = 15.9 Hz)	C-1" (134.11), C-11 (146.31) and
		C-9 (167.32)
11	7.76 (1H, *d*, *J* = 15.9 Hz)	C-9 (167.32) and C-10 (117.21)
1'	-	-
2', 6'	8.06 (2H, *m*)	C-8 (166.20) and C-4' (133.43)
3', 5'	7.46 (2H, *m*)	C-8 (166.20)
4'	7.59 (1H, *m*)	C-2' (129.83) and C-6' (129.83)
1"	-	-
2", 6"	7.53 (2H, *m*)	C-11 (146.31) and C-4" (130.66)
3", 5"	7.40 (2H, *m*)	C-1" (134.11)
4"	7.39 (1H, *m*)	-

Table 10 ^{1}H and ^{13}C NMR spectral data of compound **SAH2**

Position	δ_C [#] **(ppm)**		δ_H **(ppm)**
1	59.55	C	-
2	71.10	CH	4.25 (1H, *d*, *J* = 8.1 Hz)
3	74.40	CH	5.56 (1H, *dt*, *J* = 8.1, 2.1 Hz)
4	133.12	CH	5.86 (1H, *dt*, *J* = 9.9, 1.8 Hz)
5	124.64	CH	6.08 (1H, *ddd*, *J* = 9.9, 3.6, 2.7 Hz)
6	54.25	CH	3.59 (1H, *dd*, *J* = 3.9, 1.8 Hz)
7	62.92	CH_2	4.48 (1H, *d* (AB), *J* = 12 Hz)
			5.00 (1H, *d* (AB), *J* = 12 Hz)
8	166.20	C	-
9	167.32	C	-
10	117.21	CH	6.47 (1H, *d*, *J* = 15.9 Hz)
11	146.31	CH	7.76 (1H, *d*, *J* = 15.9 Hz)
1'	129.51	C	-
2', 6'	129.83	CH	8.06 (2H, *m*)
3', 5'	128.51	CH	7.46 (2H, *m*)
4'	133.43	CH	7.59 (1H, *m*)
1"	134.11	C	-
2", 6"	128.25	CH	7.53 (2H, *m*)
3", 5"	128.97	CH	7.40 (2H, *m*)
4"	130.66	CH	7.39 (1H, *m*)

[#] Carbon type deduced from DEPT experiment.

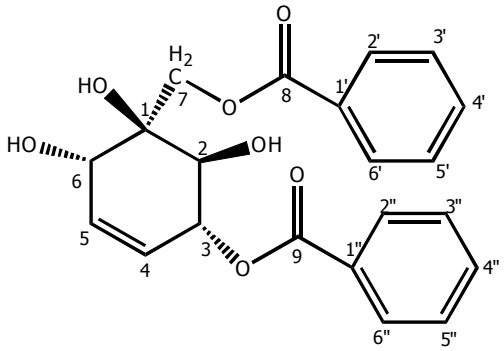
Table 11 **Comparison of ^{1}H NMR spectral data between compound** SAH2 **and** SAH1

Position	**Compound SAH2, δ_H (ppm), Recorded in $CDCl_3$**	**Compound SAH1, δ_H (ppm), Recorded in $CDCl_3$**
1	-	-
2	4.25 (1H, *d*, *J* = 8.1 Hz)	4.33 (1H, *dd*, *J* = 8, 6 Hz)
3	5.56 (1H, *dt*, *J* = 8.1, 2.1 Hz)	5.67 (1H, *ddd*, *J* = 8, 3, 2 Hz)
4	5.86 (1H, *dt*, *J* = 9.9, 1.8 Hz)	5.91 (1H, *dt*, *J* = 10, 2 Hz)
5	6.08 (1H, *ddd*, *J* = 9.9, 3.6, 2.7 Hz)	6.10 (1H, *ddd*, *J* = 10, 4, 2 Hz)
6	3.59 (1H, *dd*, *J* = 3.9, 1.8 Hz)	3.60 (1H, *dd*, *J* = 4, 2 Hz)
7	4.48 (1H, *d* (AB), *J* = 12 Hz)	4.48(1H, *d* (AB), *J* = 12 Hz)
	5.00 (1H, *d* (AB), *J* = 12 Hz)	5.00 (1H, *d* (AB), *J* = 12 Hz)
8	-	-
9	-	-
10	6.47 (1H, *d*, *J* = 15.9 Hz)	-
11	7.76 (1H, *d*, *J* = 15.9 Hz)	-
1'	-	-
2', 6'	8.06 (2H, *m*)	8.06 (2H, *m*)
3', 5'	7.46 (2H, *m*)	7.45 (2H, *m*)
4'	7.59 (1H, *m*)	7.58 (1H, *m*)
1"	-	-
2", 6"	7.53 (2H, *m*)	8.06 (2H, *m*)
3", 5"	7.40 (2H, *m*)	7.45 (2H, *m*)
4"	7.39 (1H, *m*)	7.58 (1H, *m*)

Table 12 ^{13}C NMR spectral data between compound **SAH2** and **SAH1**

Position	Compound SAH2 , δ_C (pp m)	Compound SAH1, δ_C (ppm)
1	59.55	59.49
2	71.10	71.06
3	74.40	74.82
4	133.12	132.96
5	124.64	124.73
6	54.25	54.20
7	62.92	62.91
8	166.20	166.20
9	167.32	166.87
10	117.21	-
11	146.31	-
1'	129.51	129.40
2', 6'	129.83	129.78
3', 5'	128.51	128.43
4'	133.43	133.38
1"	134.11	129.44
2", 6"	128.25	129.84
3", 5"	128.97	128.47
4"	130.66	133.44

3.1.3 Compound SAH3



Compound **SAH3** was isolated as a white solid, mp = 110-112°C, $[\alpha]_D^{25.9}$ = - 90.90° (*c* = 0.011, $CHCl_3$). The high resolution mass spectrum of this compound (Fig. 35) showed the molecular ion peak at 385.1287 *m/z* ($[M^+ +1]$), thus this compound had molecular formula $C_{21}H_{20}O_7$. The IR spectrum (Fig. 26) showed absorption bands which were ascribed to O-H stretching of hydroxyl group (3444 cm^{-1}), C=O and C-O stretching of carbonyl (1703 and 1277 cm^{-1}, respectively) and C=C stretching and C-H bending of monosubstituted phenyl ring (1601 and 709 cm^{-1}, respectively). The UV spectrum (Fig. 25) showed maxima at 230, 211 and 202 nm.

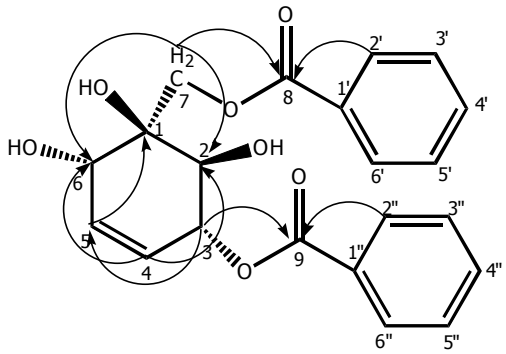
The complete analysis of ^1H and ^{13}C NMR spectral data of compound **SAH3** (see Table 16, Fig. 27 and 28) were assigned with informations provided from ^1H-^1H COSY (see Table 14, Fig. 30), ^1H-^{13}C correlation (HMQC) (Fig. 31) and ^1H-^{13}C correlation by long-range coupling (HMBC) (see Table 15, Fig. 32), along with comparison of ^1H NMR spectral data to compound **SAH1** (see Table 17). The ^{13}C NMR spectrum of compound **SAH3** recorded in $CDCl_3$ showed 17 signals for 21 carbon atoms. Analysis of the DEPT–90° and DEPT–135° spectra of this compound (see Table 13, Fig. 29) suggested the presence of one methylene carbon atom (δ 66.67), eleven signals for fifteen methine carbon atoms (δ 133 .42, 133.37, 129.81 (2xC), 129.78(2xC), 129.76, 128.41(2xC), 128.38(2xC),126.71, 74.10, 70.79 and

68.68) and five quaternary carbon atoms (δ 167.78, 167.09, 129.43, 129.23 and 75.90).

Compound **SAH3**, a derivative of compound **SAH1**, showed similar characteristic bands in IR and UV spectrum with **SAH1**. Comparison of the ^{1}H NMR spectral data (see Table 17) of the two compounds revealed close structural similarity. Differences in the spectrum of compound **SAH3** were shown as signals of hydroxyl protons at δ 3.43 (1H, *br* s) and 3.46 (1H, *br* s) which were not observed in compound SAH1. The two olefinic methine protons appeared at δ5.84 (1H-4, *ddd*, *J* = 10, 2.5, 1 Hz) and 5.99 (1H-5, *ddd*, *J* = 10, 4, 1.5 Hz) and the oxymethine protons resonating at δ4.24 (1H, *d*, *J* = 6 Hz) and 5.70 (1H, *m*) were assigned to protons at C-2 (δ 70.79) and C-3 (δ74.10), respectively. In addition, the ^{1}H NMR spectrum of compound **SAH3** showed the presence of two benzoate groups (δ7.34-7.99) and one methylene proton at δ4.72 (1H-7a, *d* (AB), *J* = 12 Hz) and 4.87 (1H-7b, *d* (AB), *J* = 12 Hz). The epoxy proton (H-6) in compound **SAH3** resonated at δ 4.34 which was lower field than H-6 (δ 3.60) of compound **SAH1** and C-6 (δ 68.68) of compound **SAH3** appeared at the lower field than C-6 (δ 54.20) of compound **SAH1**. These observations indicated that the C-1 and C-6 of compound **SAH3** should be connected to the hydroxyl group and the epoxide ring was opened.

The HMBC correlations of compound **SAH3** were the same as those of compound **SAH1**. The carbon signals at δ75.90 (C-1), 70.79 (C-2), 126.71 (C-4) and 129.76 (C-5) showed the correlation peaks with H-6 (δ4.34), indicating that the hydroxyl group was attached to C-6 (68.68). The carbon signals at δ70.79 (C-2), 126.71 (C-4), 129.76 (C-5) and 167.09 (C-9) showed correlation peaks with H-3 (δ 5.70), confirming that the benzoyl group was attached to C-3 (74.10). The carbon signals at δ 75.90 (C-1), 70.79 (C-2), 68.68 (C-6) and 167.78 (C-8) showed the correlation peaks with H-7a (δ 4.72) and H-7b (δ 4.87), indicating that the

benzoyloxymethyl group was attached to C-1 (75.90). In the NOE experiment (Fig. 33 and 34), irradiation of H-7b (δ 4.87) showed enhancement of H-7a (δ 4.72) and H-2 (δ 4.24). Irradiation of H-7a (δ 4.72) showed enhancement of H-7b (δ 4.87). Irradiation of H-6 (δ 4.34) showed enhancement of H-5 (δ 5.99). Irradiation of H-2 (δ 4.24) showed enhancement of H-7b (δ 4.87). Thus, the relative stereochemistry of this compound should be as follow : benzoyloxymethyl (at C-1), benzoyl (at C-3) and hydroxyl (at C-6) groups are on the same side and opposite side to both hydroxyl groups at C-1 and C-2.



Selected HMBC Correlation

By comparison of ^{1}H and ^{13}C NMR spectral data and specific rotation values between compound **SAH3** and **(-)-zeylenol** (see Table 19 and 20) revealed very close structural similarity. Thus, compound **SAH3** was identified as **(-)-zeylenol** which was the compound previously isolated from the roots of *Uvaria zeylanica* L. (Jolad, *et al*., 1981).

Table 13 ^{13}C and DEPT spectral data of compound **SAH3**

δ_C	**DEPT-90°**	**DEPT-135°**	**Type of carbon**
167.78			C
167.09			C
133.42	133.42	133.42	CH
133.37	133.37	133.37	CH
129.81	129.81	129.81	CH
129.78	129.78	129.78	CH
129.76	129.76	129.76	CH
129.43			C
129.23			C
128.41	128.41	128.41	CH
128.38	128.38	128.38	CH
126.71	126.71	126.71	CH
75.90			C
74.10	74.10	74.10	CH
70.79	70.79	70.79	CH
68.68	68.68	68.68	CH
66.67		66.67	CH_2

Table 14 500 MHz COSY correlation of some protons of **SAH3**

δ_H (ppm)	Proton correlation with δ_H (ppm)
H-2 (4.24)	H-3 (5.70)
H-3 (5.70)	H-2 (4.24), H-4 (5.84), H-5 (5.99)
H-4 (5.84)	H-3 (5.70), H-5 (5.99), H-6 (4.34)
H-5 (5.99)	H-3 (5.70), H-4 (5.84), H-6 (4.34)
H-6 (4.34)	H-5 (5.99)
H-7a (4.72)	H-7b (4.87)
H-2', H-6' (7.94)	H-3', H-5' (7.37)
H-2", H-6" (7.99)	H-3", H5" (7.37)
H-3', H-5' (7.37)	H-2', H-6' (7.94), H-4'(7.52)
H-3", H5" (7.37)	H-2", H-6" (7.99), H-4" (7.52)
H-4' (7.52)	H-3', H-5' (7.37)
H-4" (7.52)	H-3", H5" (7.37)

Table 15 Major HMBC correlation of compound **SAH3**

Position	δ_H (ppm)	δ_C (ppm)
1	-	-
2	4.24 (1H, *d*, *J* = 6 Hz)	C-3 (74.10), C-4 (126.71) and C-6 (68.68)
3	5.70 (1H, *m*)	C-2 (70.79), C-4 (126.71), C-5 (129.76) and C-9 (167.09)
4	5.84 (1H, *ddd*, *J* = 10, 2.5, 1 Hz)	C-2 (70.79), C-3 (74.10), C-5 (129.76) and C-6 (68.68)

Table 15 (Continued)

Position	δ_H (ppm)	δ_C (ppm)
5	5.99 (1H, *ddd*, *J* = 10, 4, 1.5 Hz)	C-1 (75.90), C-3 (74.10) and C-4 (126.71)
6	4.34 (1H, *d*, *J* = 4 Hz)	C-1 (75.90), C-2 (70.79), C-4 (126.71) and C-5 (129.76)
7	4.72 (1H, *d* (AB), *J* = 12 Hz) 4.87 (1H, *d* (AB), *J* = 12 Hz)	C-1 (75.90), C-2 (70.79), C-6 (68.68) and C-8 (167.78)
8	-	-
9	-	-
1'	-	-
1"	-	-
2', 6'	7.94 (2H, *m*)	C-3' , C-5' (128.41), C-1' (129.23), C-4' (133.42) and C-8 (167.78)
2", 6"	7.99 (2H, *m*)	C-3",C-5" (128.38), C-1" (129.43), C-4" (133.37) and C-9 (167.09)
3', 5' / 3", 5"	7.37 (4H, *m*)	C-2', C-6' (129.78) and C-4' (133.42) / C-2", C-6" (129.81) and C-4"(133.37)
4' / 4"	7.52 (2H, *m*)	C-2', C-6' (129.78) and C-3', C-5' (128.41) / C-2", C-6" (129.81) and C3", C-5" (128.38)
-OH	3.17(1H, *br s*)	-
-OH	3.43(1H, *br s*)	-
-OH	3.46 (1H, *br s*)	-

Table 16 ^{1}H and ^{13}C NMR spectral data of compound **SAH3**

Position	δ_C [#] (ppm)		δ_H (ppm)
1	75.90	C	-
2	70.79	CH	4.24 (1H, *d*, *J* = 6 Hz)
3	74.10	CH	5.70 (1H, *m*)
4	126.71	CH	5.84 (1H, *ddd*, *J* = 10, 2.5, 1 Hz)
5	129.76	CH	5.99 (1H, *ddd*, *J* = 10, 4, 1.5 Hz)
6	68.68	CH	4.34 (1H, *d*, *J* = 4 Hz)
7	66.67	CH_2	4.72 (1H, *d* (AB), *J* = 12 Hz)
			4.87 (1H, *d* (AB), *J* = 12 Hz)
8	167.78	C	-
9	167.09	C	-
1' [a]	129.23	C	-
2', 6' [b]	129.78	CH	7.94 (2H, *m*)
3', 5' [c]	128.41	CH	7.37 (2H, *m*)
4' [d]	133.42	CH	7.52 (1H, *m*)
1" [a]	129.43	C	-
2", 6" [b]	129.81	CH	7.99 (2H, *m*)
3", 5" [c]	128.38	CH	7.37 (2H, *m*)
4" [d]	133.37	CH	7.52(1H, *m*)
-OH	-	-	3.17 (1H, *br s*)
-OH	-	-	3.43 (1H, *br s*)
-OH	-	-	3.46 (1H, *br s*)

[#] Carbon type deduced from DEPT experiment.

[a, b, c, d] May be interchangeable.

Table 17 Comparison of ^{1}H NMR spectral data between compound **SAH3** and **SAH1**

Position	**Compound SAH3, δ_H (ppm), Recorded in $CDCl_3$**	**Compound SAH1, δ_H (ppm), Recorded in $CDCl_3$**
1	-	-
2	4.24 (1H, *d*, *J* = 6 Hz)	4.33 (1H, *dd*, *J* = 8, 2 Hz)
3	5.70 (1H, *m*)	5.67 (1H, *ddd*, *J* = 8, 3, 2 Hz)
4	5.84 (1H, *ddd*, *J* = 10, 2.5, 1 Hz)	5.91 (1H, *ddd*, *J* = 10, 4, 2 Hz)
5	5.99 (1H, *ddd*, *J* = 10, 4, 1.5 Hz)	6.10 (1H, *ddd*, *J* = 10, 4, 2 Hz)
6	4.34 (1H, *d*, *J* = 4 Hz)	3.60 (1H, *dd*, *J* = 4, 2 Hz)
7	4.72 (1H, *d* (AB), *J* = 12 Hz)	4.48(1H, *d* (AB), *J* = 12 Hz)
	4.87 (1H, *d* (AB), *J* = 12 Hz)	5.00 (1H, *d* (AB), *J* = 12 Hz)
8	-	-
9	-	-
1'	-	-
2', 6' / 2", 6"	7.94 (2H, *m*) / 7.99 (2H, *m*)	8.06 (4H, *m*)
3', 5' / 3", 5"	7.37 (4H, *m*)	7.45 (4H, *m*)
4' / 4"	7.52 (2H, *m*)	7.58 (2H, *m*)
1"	-	-
-OH	3.17 (1H, *br s*)	3.17 (1H, *br s*)
-OH	3.43 (1H, *br s*)	-
-OH	3.46 (1H, *br s*)	-

Table 18 Comparison of ^{13}C NMR spectral data between compound **SAH3** and **SAH1**

Position	Compound SAH3, δ_C (ppm), Recorded in $CDCl_3$	Compound SAH1, δ_C (ppm), Recorded in $CDCl_3$
1	75.90	59.49
2	70.79	71.06
3	74.10	74.82
4	126.71	132.96
5	129.76	124.73
6	68.68	54.20
7	66.67	62.91
8	167.78	166.20
9	167.09	166.87
1' [a]	129.23	129.40
2', 6' [b]	129.78	129.78
3', 5' [c]	128.41	128.43
4' [d]	133.42	133.38
1" [a]	129.43	129.44
2", 6" [b]	129.81	129.84
3", 5" [c]	128.38	128.47
4" [d]	133.37	133.44

[a, b, c, d] May be interchangeable.

Table 19 Comparison of ^{1}H NMR spectral data between compound **SAH3** and **(-)-zeylenol**

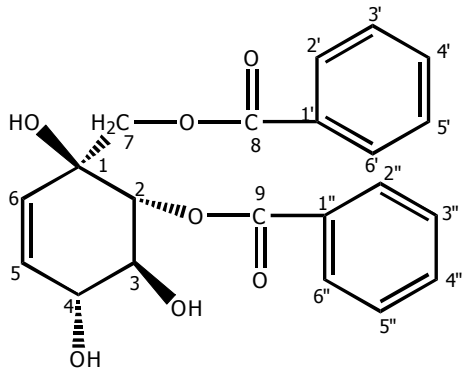
Position	SAH3, δ_H (ppm)	(-)-Zeylenol, δ_H (ppm)
1	-	-
2	4.24 (1H, *d*, *J* = 6 Hz)	4.22 (1H, *d*, *J* = 6.1 Hz)
3	5.70 (1H, *m*)	5.70 (1H, *dddd*, *J* = 6.1, 2.6, 1.6, 1.1 Hz)
4	5.84 (1H, *ddd*, *J* = 10, 2.5, 1 Hz)	5.88 (1H, *ddd*, *J* = 10.1, 2.6, 0.7Hz)
5	5.99 (1H, *ddd*, *J* = 10, 4, 1.5 Hz)	5.99 (1H, *ddd, J* = 10.1, 4, 1.6 Hz)
6	4.34 (1H, *d*, *J* = 4 Hz)	4.32 (1H, *ddd*, *J* = 4, 1.1, 0.7 Hz)
7	4.72 (1H, *d* (AB), *J* = 12 Hz)	4.75 (1H, *d* (AB), *J* = 12.3 Hz)
	4.87 (1H, *d* (AB), *J* = 12 Hz)	4.89 (1H, *d* (AB), *J* = 12.3 Hz)
8	-	-
9	-	-
1',1"	-	-
2', 6' / 2", 6"	7.94 (2H, *m*) / 7.99 (2H, *m*)	7.89 (2H, *m*) / 8.02 (2H, *m*)
3', 5' / 3", 5"	7.37 (4H, *m*)	7.40 (4H, *m*)
4' / 4"	7.52 (2H, *m*)	7.55 (2H, *m*)
-OH	3.17 (1H, *br s*)	3.18 (1H, *s*)
-OH	3.43 (1H, *br s*)	2.96 (1H, *s*)
-OH	3.46 (1H, *br s*)	3.32 (1H, *s*)

Table 20 Comparison of ^{13}C NMR spectral data between compound **SAH3** and **(-)-zeylenol**

Position	**SAH3, δ_C (ppm)**	**(-)-Zeylenol, δ_C (ppm)**
1	75.90	76.0
2	70.79	68.7
3	74.10	74.4
4	126.71	127.0
5	129.76	129.5
6	68.68	70.9
7	66.67	66.8
8	167.78	165.0
9	167.09	165.0
1' [a]	129.23	128.5
2', 6' [b]	129.78	129.9
3', 5' [c]	128.41	128.5
4' [d]	133.42	133.5
1" [a]	129.43	128.5
2", 6" [b]	129.81	129.9
3", 5" [c]	128.38	128.5
4" [d]	133.37	133.5

[a, b, c, d] May be interchangeable.

3.1.4 Compound SAC1

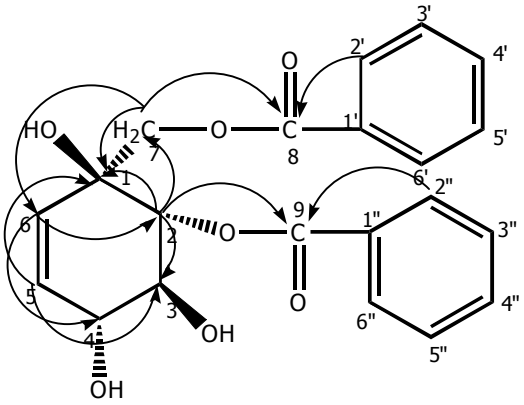


Compound **SAC1** was isolated as a white solid, mp = 149-151°C. Its UV absorption spectrum (Fig. 36) showed maxima at 230, 211 and 203 nm. The IR spectrum of compound **SAC1** (Fig. 37) showed absorption bands at 3453 cm^{-1} (hydroxyl group), 1702 cm^{-1} (carbonyl group), 1273 (C-O stretching) and 709 cm^{-1} (monosubstituted phenyl ring).

The complete analysis of ^{13}C and ^{1}H NMR spectrum of compound **SAC1** (see Table 24 , Fig. 38 and 39) were assigned with the informations provided from ^{1}H-^{1}H COSY (see Table 22 , Fig. 41), HMQC (Fig.42) and HMBC (see Table 23 , Fig. 43). The ^{13}C NMR spectrum of compound **SAC1** recorded in a mixture of $CDCl_3$ in acetone (see Table 21, Fig. 39) showed 14 signals for 21 carbon atoms. The DEPT-135° (see Table 21, Fig. 40) indicating the existence of one methylene carbon atom (δ 66.97), eleven signals for fifteen methine carbon atoms (δ 133.91 (2xC) , 133.31 (2xC), 130.23, 129.99, 129.69 (2xC), 129.37 (2xC), 128.65, 128.48, 79.64, 73.95 and 72.24) and two signals for three quaternary carbon atoms (δ 128.78 (2xC) and 74.31).

Compound **SAC1** showed the same characteristic peak in the IR and UV spectrum of compound **SAH3**. Comparison of the ^{1}H NMR spectrum (see Table 25) of the two compounds revealed the typical cyclohexene oxide. Differences in the spectrum of compound **SAC1** was shown as a doublet signal of the hydroxyl protons at δ 2.65 (1H, *br d*, *J* = 4.5 Hz) and 2.56 (1H, *br d*, *J* = 4.2 Hz), a singlet signal of the hydroxyl proton at δ 3.80 (1H, *s*) which were assigned to 3-OH, 4-OH and 1-OH, respectively. Three oxymethine protons resonated at δ 5.33 (1H, *d*, *J* = 10.5 Hz), 4.16 (1H, *ddd*, *J* = 10.5, 7.5, 4.2 Hz) and 4.41 (1H, *ddd*, *J* = 10.2, 4.5, 2.1 Hz) which were assigned to H-2, H-3 and H-4, respectively. Two olefinic protons at C-5 and C-6 positions appeared at δ 5.91 (1H, *dd*, *J* = 10.5, 2.1 Hz) and 5.75 (1H, *dd*, *J* = 10.2, 2.1 Hz), respectively.

The correlation peaks in the HMBC spectra of H-7a (δ 4.46) and H-7b (δ 4.51) with the carbons at δ 166 (C-8), 129.37 (C-6) and 74.31 (C-1); of H-6 (δ 5.75) with the carbons at δ 79.64 (C-2) and 72.24 (C-4); of H-5 with the carbons at δ 74.31 (C-1) and 72.24 (C-4), indicating that the benzoyloxymethyl group was attached to C-1 (74.31) and the double bond was formed between C-5 (130.23) and C-6 (129.37). The correlation peaks between H-2 (δ 5.33) with the carbons at δ 168 (C-9), 74.31 (C-1), 73.95 (C-3) and 66.97 (C-7), confirming that the benzoyl group was connected to C-2 (79.64). From NOESY experiment (Fig. 44), 2H-7 (δ 4.46 and 4.51) showed the cross peak with H-3 (δ 4.16); H-4 (δ 4.41) showed the cross peak with H-2 (δ 5.33). Thus, this observation confirming that the benzoyloxymethyl (at C-1), benzoyl (at C-2) and the hydroxy (at C-4) groups are on the same side and opposite side to both hydroxyl groups at C-1 and C-3. Compound **SAC1** has not been reported before. The structure of this compound was finally confirmed by X-ray diffraction. (see Fig. 2)



Selected HMBC Correlation

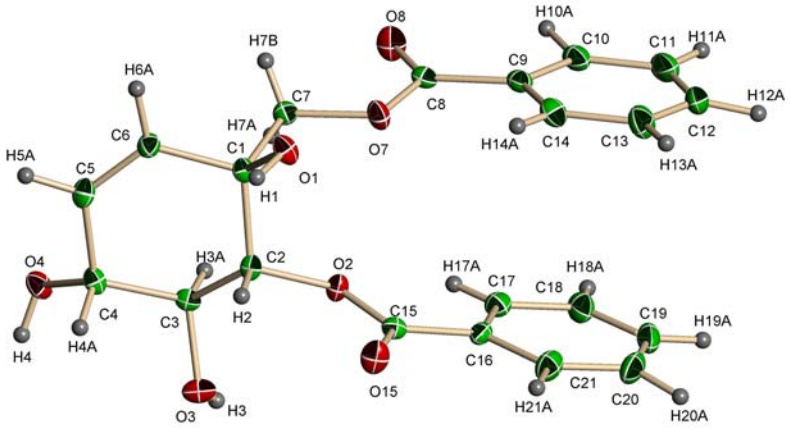


Figure 2 X-ray ORTEP diagram of compound **SAC1**

Table 21 ^{13}C and DEPT spectral data of compound **SAC1**

δ_C	**DEPT-135°**	**Type of carbon**
133.91	133.90	CH
133.31	133.30	CH
130.23	130.22	CH
129.99	129.98	CH
129.69	129.68	CH
129.37	129.35	CH
128.78		C
128.65	128.64	CH
128.48	128.47	CH
79.64	79.63	CH
74.31		C
73.95	73.94	CH
72.24	72.22	CH
66.97	66.96	CH_2

Table 22 300 MHz COSY correlation of some protons of compound **SAC1**

δ_H (ppm)	Proton correlation with δ_H (ppm)
H-2 (5.33)	H-3 (4.16)
H-3 (4.16)	H-2 (5.33), H-4 (4.41), 3-OH (2.65)
H-4 (4.41)	H-3 (4.16), 4-OH (2.56)
H-5 (5.91)	H-6 (5.75)
H-7a (4.46)	H-7b (4.51)
3-OH (2.65)	H-3 (4.16)
4-OH (2.56)	H-4 (4.41)
H-2', H-6' (7.96)	H-3', H-5' (7.37), H-4' (7.53)
H-2", H-6" (8.03)	H-3", H5" (7.42), H-4" (7.57)
H-3', H-5' (7.37)	H-2', H-6' (7.96), H-4' (7.53)
H-3", H5" (7.42)	H-2", H-6" (8.03), H-4" (7.57)
H-4' (7.53)	H-2', H-6' (7.96) ,H-3', H-5'(7.37)
H-4" (7.57)	H-2", H-6" (8.03), H-3", H5" (7.42)

Table 23 Major HMBC correlation of compound **SAC1**

Position	δ_H (ppm)	δ_C (ppm)
1	-	-
2	5.33 (1H, *d*, *J* = 10.5 Hz)	C-1 (74.31), C-3(73.95), C-7 (66.97) and C-9 (168)
3	4.16 (1H, *ddd, J* = 10.5, 7.5, 4.2 Hz)	-
4	4.41 (1H, *ddd*, *J* = 10.2, 4.5, 2.1 Hz)	C-3 (73.95)
5	5.91 (1H, *dd*, *J* = 10.5, 2.1 Hz)	C-1 (74.31) and C-3 (73.95)
6	5.75 (1H, *dd*, *J* = 10.2, 2.1 Hz)	C-2 (79.64) and C-4 (72.24)
7	4.46 (1H, *d* (AB), *J* = 11.7 Hz) 4.51 (1H, *d* (AB), *J* = 11.7 Hz)	C-1 (74.31), C-6 (129.37) and C-8 (166)
8	-	-
9	-	-
1'	-	-
2', 6'	7.96 (2H, *dd, J* = 7.8, 0.6 Hz)	C-4' (133.31) and C-8 (166)
3',5'	7.37 (2H, *m*)	C-1' (128.78)
4'	7.53 (1H, *m*)	C-2', C-6' (129.69)
1"	-	-
2", 6"	8.03 (2H, *dd*, *J* = 7.8, 0.6 Hz)	C-4" (133.91) and C-9 (168)
3",5"	7.42 (2H, *m*)	C-1" (128.78)
4"	7.57 (1H, *m*)	C-2", C-6" (129.99)
1-OH	3.80 (1H, *s*)	-
3-OH	2.65 (1H, *br d*, *J* = 4.5 Hz)	-
4-OH	2.56 (1H, *br d*, *J* = 4.2 Hz)	-

Table 24 ^{1}H and ^{13}C NMR spectra data of compound **SAC1**

Position	δ_C [#] (ppm)		δ_H (ppm)
1	74.31	C	-
2	79.64	CH	5.33 (1H, *d*, *J* = 10.5 Hz)
3	73.95	CH	4.16 (1H, *ddd, J* = 10.5, 7.5, 4.2 Hz)
4	72.24	CH	4.41 (1H, *ddd*, *J* = 10.2, 4.5, 2.1 Hz)
5	130.23	CH	5.91 (1H, *dd*, *J* = 10.5, 2.1 Hz)
6	129.37	CH	5.75 (1H, *dd*, *J* = 10.2, 2.1 Hz)
7	66.97	CH_2	4.46 (1H, *d* (AB), *J* = 11.7 Hz)
			4.51 (1H, *d* (AB), *J* = 11.7 Hz)
8	166	C	-
9	168	C	-
1'	128.78	C	-
2', 6'	129.69	CH	7.96 (2H, *dd, J* = 7.8, 0.6 Hz)
3',5'	128.65	CH	7.37 (2H, *m*)
4'	133.31	CH	7.53 (1H, *m*)
1"	128.78	C	-
2", 6"	129.99	CH	8.03 (2H, *dd*, *J* = 7.8, 0.6 Hz)
3",5"	128.48	CH	7.42 (2H, *m*)
4"	133.91	CH	7.57 (1H, *m*)
1-OH	-	-	3.80 (1H, *s*)
3-OH	-	-	2.65 (1H, *br d*, *J* = 4.5 Hz)
4-OH	-	-	2.56 (1H, *br d*, *J* = 4.2 Hz)

[#] Carbon type deduced from DEPT experiment.

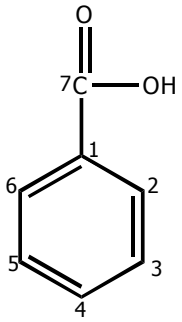
Table 25 Comparison of ^{1}H NMR spectral data between compound **SAC1** and **SAH3**

Position	Compound SAC1, δ_H (ppm), Recorded in $CDCl_3$	Compound SAH3, δ_H (ppm), Recorded in $CDCl_3$
1	-	-
2	5.33 (1H, *d*, *J* = 10.5 Hz)	4.24 (1H, *d*, *J* = 6 Hz)
3	4.16 (1H, *ddd, J* = 10.5, 7.5, 4.2 Hz)	5.70 (1H, *m*)
4	4.41 (1H, *ddd*, *J* = 10.2, 4.5, 2.1 Hz)	5.84 (1H, *ddd*, *J* = 10, 2.5, 1 Hz)
5	5.91 (1H, *dd*, *J* = 10.5, 2.1 Hz)	5.99 (1H, *ddd*, *J* = 10, 4, 1.5 Hz)
6	5.75 (1H, *dd*, *J* = 10.2, 2.1 Hz)	4.34 (1H, *d*, *J* = 4 Hz)
7	4.46 (1H, *d* (AB), *J* = 11.7 Hz)	4.72 (1H, *d* (AB), *J* = 12 Hz)
	4.51 (1H, *d* (AB), *J* = 11.7 Hz)	4.87 (1H, *d* (AB), *J* = 12 Hz)
8	-	-
9	-	-
1'	-	-
2', 6'	7.96 (2H, *dd, J* = 7.8, 0.6 Hz)	7.94 (2H, *m*)
3',5'	7.37 (2H, *m*)	7.37 (2H, *m*)
4'	7.53 (1H, *m*)	7.52 (1H, *m*)
1"	-	-
2", 6"	8.03 (2H, *dd*, *J* = 7.8, 0.6 Hz)	7.99 (2H, *m*)
3",5"	7.42 (2H, *m*)	7.37 (2H, *m*)
4"	7.57 (1H, *m*)	7.52(1H, *m*)
-OH	3.80 (1H, *s*)	3.17 (1H, *br s*)
-OH	2.65 (1H, *br d*, *J* = 4.5 Hz)	3.43 (1H, *br s*)
-OH	2.56 (1H, *br d*, *J* = 4.2 Hz)	3.46 (1H, *br s*)

Table 26 Comparison of ^{13}C NMR spectral data between compound **SAC1** and **SAH3**

Position	Compound SAC1, δ_C (ppm), Recorded in $CDCl_3$	Compound SAH3, δ_C (ppm), Recorded in $CDCl_3$
1	74.31	75.90
2	79.64	70.79
3	73.95	74.10
4	72.24	126.71
5	130.23	129.76
6	129.37	68.68
7	66.97	66.67
8	166	167.78
9	168	167.09
1'	128.78	129.23
2', 6'	129.69	129.78
3',5'	128.65	128.41
4'	133.31	133.42
1"	128.78	129.43
2", 6"	129.99	129.81
3",5"	128.48	128.38
4"	133.91	133.37

3.1.5 Compound SAM1

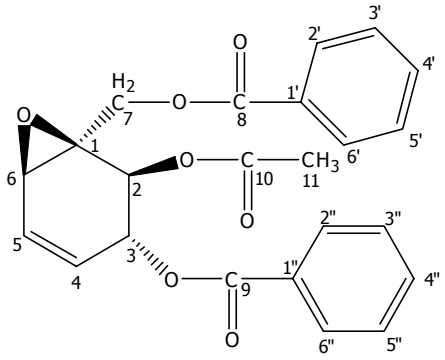


Compound **SAM1** was isolated as a white solid, mp. = 117-118 ^{o}C. The UV spectrum (Fig. 46) showed maximum absorptions at 227, 211 and 202 nm. The IR spectrum (Fig. 47) showed the absorption bands at 3500-2500 cm^{-1} (O-H stretching), 1686 cm^{-1} (C=O stretching), 1292 cm^{-1} (C-O stretching), 1686 and 705 cm^{-1} (C=C stretching and C-H bending of monosubstituted phenyl ring). The ^{1}H NMR spectrum of this compound (Fig. 48) showed a doublet of doublet of *ortho*-aromatic protons at δ 8.13 (2H, *dd*, *J* = 7.8, 1.5 Hz), a triplet of triplet of *para*-aromatic proton at δ 7.64 (1H, *tt*, *J* = 7.5, 1.2 Hz) and a triplet of *meta*-aromatic protons at δ 7.48 (2H, *t*, *J* = 7.8 Hz). Thus compound **SAM1** was identified as benzoic acid.

3.2 Structural elucidation of compound synthesized from pipoxide : compound SAH1, and compound SAH2.

The acetylation, hydrobromination, hydrolysis and epoxidation reactions by using pipoxide as a starting material afforded four compounds : pipoxide acetate **(ST1)**, pipoxide bromohydrin **(ST2)**, zeylenol **(ST3)** and diepoxide **(ST4)**. The acetylation of cinnamoxide **(SAH2)** yielded one compound, cinnamoxide acetate **(ST5)**. Their structures were determined by 1D and/or 2D NMR spectroscopic data.

3.2.1 Compound ST1



Acetylation reaction of compound **SAH1**, pipoxide, with acetyl chloride in methylene chloride in the presence of trimethylamine at room temperature under nitrogen atmosphere for 40 hrs gave compound **ST1** and unreacted starting material after PLC separation (silica gel, 25 % EtOAc : hexane). Compound **ST1** was a colorless viscous liquid, $[\alpha]_D^{25.9}$ = + 14.08 ^{o} (c = 0.071, $CHCl_3$). The UV absorption spectrum (Fig. 49) showed maxima at 230, 212 and 203 nm and the IR spectrum

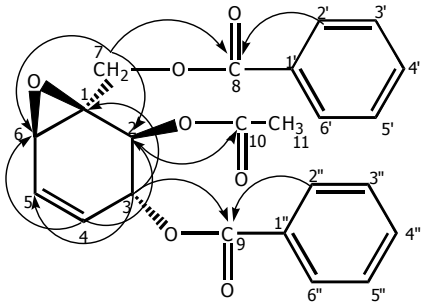
(Fig. 50) showed absorption bands at 1749 and 1720 cm^{-1} corresponding to the carbonyl groups and 1598, 705 and 700 cm^{-1} corresponding to the monosubstituted phenyl ring. The molecular formula (Fig. 59) was determined as $C_{23}H_{20}O_7$ by EITOFMS ([M^++1], *m/z* 409).

The complete analysis of ^{13}C and ^{1}H NMR spectrum of compound **ST1** (see Table 30, Fig.51 and 52) were assigned with the informations provided from ^{1}H-^{1}H COSY (see Table 28, Fig. 54), HMQC (Fig. 55), HMBC (see Table 29, Fig. 56) and NOE experiments (Fig. 57 and 58). The ^{13}C NMR spectrum of compound **ST1** (see Table 27, Fig. 52) recorded in $CDCl_3$ showed 17 signals for 23 carbons. Analysis of the DEPT–90° and DEPT–135° spectrum of this compound (see Table 27, Fig. 53) suggested the presence of one methyl carbon (δ 20.74), one methylene carbon (δ 62.20), ten signals for fifteen methine carbons (δ 133.55, 133.44, 133.39, 129.82 (2xC), 129.79 (2xC), 128.53 (4xC), 124.26, 72.13, 71.02 and 54.54) and five signals for six quaternary carbons (δ 170.19, 165.83, 129.40, 129.30 and 58.37).

Compound **ST1**, a derivative of compound **SAH1**, showed the same characteristic peak in the IR and UV spectrum with compound **SAH1**. Comparison of the ^{1}H NMR spectrum (see Table 31) of the two compounds revealed their close structural similarity. Differences in the spectrum of compound **ST1** was shown as an acetoxy proton at δ 2.08 (3H, s) which was not observed in compound **SAH1** The methylene proton at C-7 appeared as an AB system at δ 4.42 (1H, *d*, *J* = 12 Hz) and 4.68 (1H, *d*, *J* = 12 Hz). The epoxy-methine proton (H-6), and two olefinic methine protons (H-4 and H-5) appeared at δ 3.63 (1H, *dd*, *J* = 3.9, 1.8 Hz), 6.12 (1H, *dt*, *J* = 9.9, 3.3 Hz) and 5.95 (1H, *dt*, *J* = 9.9, 1.8 Hz), respectively. The two benzoyl groups showed a multiplet signal at δ 8.03 (4H, *m*), 7.45 (4H, *m*) and 7.58 (2H, *m*). The oxymethine protons resonated at δ 5.77 (1H, *dt*, *J* = 8.4, 2.1 Hz) and 5.89 (1H, *d*, *J* = 8.4 Hz) could be assigned to H-3 and H-2, respectively. The H-2 (δ 5.89, 1H, *d*, *J* = 8.4 Hz) of compound **ST1** appeared at the lower field than H-2 of compound **SAH1**

(δ 4.33, 1H, *dd*, *J* = 8, 2 Hz). These observations indicated that the C-2 position should be connected with the acetyl group.

The HMBC correlation of compound **ST1** (see Table 29, Fig. 56) were similar to compound **SAH1** except H-2 of **ST1** (δ 5.89) showed correlation peaks with C-3 (72.13), C-6 (54.54) and C-10 (170.19), thus confirmed the position of the acetoxy group at C-2 (71.02). These observations indicated that both compounds are derivatives. From NOE experiment, irradiation of H-7b (δ 4.68) resulted in the enhancement of the signals at H-7a (δ 4.42) and H-2 (δ 5.89). Irradiation of H-7a (δ 4.42) resulted in the enhancement of the signals at H-7b (δ 4.68) and H-6 (δ 3.63). Irradiation of H-6 (δ 3.63) resulted in the enhancement of the signals at H-7a (δ 4.42) and H-4 (δ 6.12). Irradiation H-3 (δ 5.77) resulted in the enhancement of the signal at H-5 (δ 5.95). Irradiation of H-2 (δ 5.89) resulted in the enhancement of the signals at H-7b (δ 4.68). Thus, this compound should be β-acetoxyl group at C-2.



Selected HMBC Correlation

By comparison of ^{1}H and ^{13}C NMR spectral data between compound **ST1** and **pipoxide acetate** (see Table 33), both compounds showed similarity. The relative stereochemistry of compound **ST1** was deduced with optical rotation $[\alpha]_D^{25.9}$ = + 14.08^{o} (*c* = 0.071 g/100 cm^{-3}, $CHCl_3$), this being identical to **pipoxide acetate** value, $[\alpha]_D^{25.9}$ = + 9^{o} (*c* = 4.28, $CHCl_3$) (Kodpinid, 1984). Thus compound **ST1** was identified as

pipoxide acetate which was previously synthesized from pipoxide (44) (Kodpinid, 1984).

Table 27 ^{13}C and DEPT spectral data of compound **ST1**

δ_C	**DEPT-135°**	**Type of carbon**
170.19		C
165.83		C
133.55	133.55	CH
133.44	133.44	CH
133.39	133.39	CH
129.79	129.79	CH
129.82	129.83	CH
129.40		C
129.30		C
128.53	128.53	CH
124.26	124.27	CH
71.02	71.02	CH
72.13	72.13	CH
62.20	62.20	CH_2
58.37		C
54.54	54.54	CH
20.74	20.74	CH_3

Table 28 300 MHz COSY correlation of some protons of compound **ST1**

δ_H (ppm)	**Proton correlation with δ_H (ppm)**
H-2 (5.89)	H-3 (5.77)
H-4 (6.12)	H-5 (5.95), H-6 (3.63)
H-5 (5.95)	H-4 (6.12)
H-6 (3.63)	H-4 (6.12)
H-7a (4.42)	H-7b (4.68)
H-2', H-6' (8.03)	H-3', H-5' (7.45)
H-2", H-6" (8.03)	H-3", H5" (7.45)
H-3', H-5' (7.45)	H-2', H-6' (8.03), H-4' (7.58)
H-3", H5" (7.45)	H-2", H-6" (8.03), H-4" (7.58)
H-4' (7.58)	H-3', H-5' (7.45)
H-4" (7.58)	H-3", H5" (7.45)

Table 29 Major HMBC correlation of compound **ST1**

Position	**δ_H (ppm)**	**δ_C (ppm)**
1	-	-
2	5.89 (1H, *d*, *J* = 8.4 Hz)	C-3 (72.13), C-6 (54.54) and C-10 (170.19)
3	5.77 (1H, *dt*, *J* = 8.4, 2.1 Hz)	C-1 (58.37), C-2 (71.02), C-4 (124.26), C-5 (133.55) and C-9 (165.83)

Table 29 (Continued)

Position	δ_H (ppm)	δ_C (ppm)
1	-	-
2	5.89 (1H, *d*, *J* = 8.4 Hz)	C-3 (72.13), C-6 (54.54) and C-10 (170.19)
3	5.77 (1H, *dt*, *J* = 8.4, 2.1 Hz)	C-1 (58.37), C-2 (71.02), C-4 (124.26), C-5 (133.55) and C-9 (165.83)
4	6.12 (1H, *dt*, *J* = 9.9, 3.3 Hz)	C-2 (71.02) and C-6 (54.54)
5	5.95 (1H, *dt*, *J* = 9.9, 1.8 Hz)	C-3 (72.13)
6	3.63 (1H, *dd*, *J* = 3.9, 1.8 Hz)	C-4 (124.26) and C-5 (133.55)
7	4.42 (1H, *d*, *J* = 12 Hz) 4.68 (1H, *d*, *J* = 12 Hz)	C-1 (58.37), C-2 (71.02), C-6 (54.54) and C-8 (165.83)
8	-	-
9	-	-
10	-	-
11	2.08 (3H, *s*)	C-2 (71.02) and C-10 (170.19)
1'	-	-
2', 6'	8.03 (2H, *m*)	C-4' (133.39) and C-8 (165.83)
3',5'	7.45 (2H, *m*)	C-8 (165.83)
4'	7.58 (1H, *m*)	C-2', C-6' (129.82)
1"	-	-
2", 6"	8.03 (2H, *m*)	C-4" (133.44) and C-9 (165.83)
3",5"	7.45 (2H, *m*)	C-9 (165.83)

4"	7.58 (1H, *m*)	C-2", C-6" (129.79)

Table 30 ^{1}H and ^{13}C NMR spectral data of compound **ST1**

Position	δ_C [#] **(ppm)**		δ_H **(ppm)**
1	58.37	C	-
2	71.02	CH	5.89 (1H, *d*, *J* = 8.4 Hz)
3	72.13	CH	5.77 (1H, *dt*, *J* = 8.4, 2.1 Hz)
4	124.26	CH	6.12 (1H, *dt*, *J* = 9.9, 3.3 Hz)
5	133.55	CH	5.95 (1H, *dt*, *J* = 9.9, 1.8 Hz)
6	54.54	CH	3.63 (1H, *dd*, *J* = 3.9, 1.8 Hz)
7	62.20	CH_2	4.42 (1H, *d, J* = 12 Hz)
			4.68 (1H, *d, J* = 12 Hz)
8	165.83	C	-
9	165.83	C	-
10	170.19	C	-
11	20.74	CH_3	2.08 (3H, *s*)
1' [a]	129.30	C	-
2', 6' [b]	129.82	CH	8.03 (2H, *m*)
3',5'	128.53	CH	7.45 (2H, *m*)
4' [c]	133.39	CH	7.58 (1H, *m*)
1" [a]	129.40	C	-
2", 6" [b]	129.79	CH	8.03 (2H, *m*)
3",5"	128.53	CH	7.45 (2H, *m*)
4" [c]	133.44	CH	7.58 (1H, *m*)

[#] Carbon type deduced from DEPT experiment.

[a, b, c] May be interchangeable.

Table 31 Comparison of ^{1}H NMR spectral data between compound **ST1** and **SAH1**

Position	Compound ST1, δ_H (pp m)	Compound SAH1, δ_H (pp m)
1	-	-
2	5.89 (1H, *d*, *J* = 8.4 Hz)	4.33 (1H, *dd*, *J* = 8, 6 Hz)
3	5.77 (1H, *dt*, *J* = 8.4, 2.1 Hz)	5.67 (1H, *ddd*, *J* = 8, 3, 2 Hz)
4	6.12 (1H, *dt*, *J* = 9.9, 3.3 Hz)	5.91 (1H, *dt*, *J* = 10, 2 Hz)
5	5.95 (1H, *dt*, *J* = 9.9, 1.8 Hz)	6.10 (1H, *ddd*, *J* = 10, 4, 2 Hz)
6	3.63 (1H, *dd*, *J* = 3.9, 1.8 Hz)	3.60 (1H, *dd*, *J* = 4, 2 Hz)
7	4.42 (1H, *d* (AB), *J* = 12 Hz)	4.48 (1H, *d* (AB), *J* = 12 Hz) ,
	4.68 (1H, *d* (AB), *J* = 12 Hz)	5.00 (1H, *d* (AB), *J* = 12 Hz)
8	-	-
9	-	-
10	-	-
11	2.08 (3H, *s*)	-
1' / 1"	-	-
2', 6' / 2", 6"	8.03 (4H, *m*)	8.06 (4H, *m*)
3',5' / 3",5"	7.45 (4H, *m*)	7.45 (4H, *m*)
4' / 4"	7.58 (2H, *m*)	7.58 (2H, *m*)

Table 32 Comparison of ^{13}C spectral data between compound **ST1** and **SAH1**

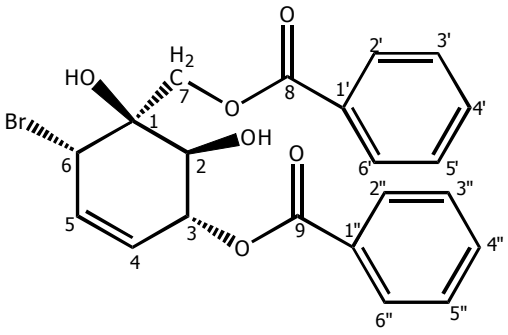
Position	Compound ST1, δ_C (ppm)	Compound SAH1, δ_C (ppm)
1	58.37	59.49
2	71.02	71.06
3	72.13	74.82
4	124.26	132.96
5	133.55	124.73
6	54.54	54.20
7	62.20	62.91
8	165.83	166.20
9	165.83	166.87
10	170.19	-
11	20.74	-
1' [a]	129.30	129.40
2', 6' [b]	129.82	129.78
3',5'	128.53	128.43
4' [c]	133.39	133.38
1" [a]	129.40	129.44
2", 6" [b]	129.79	129.84
3",5"	128.53	128.47
4" [c]	133.44	133.44

[a, b, c] May be interchangeable.

Table 33 Comparison of ^{1}H NMR spectral data between compound **ST1** and **pipoxide acetate**

Position	ST1, δ_H (ppm)	Pipoxide acetate, δ_H (ppm)
1	-	-
2	5.89 (1H, *d*, *J* = 8.4 Hz)	5.69 (1H, *d*, *J* = 8 Hz)
3	5.77 (1H, *dt*, *J* = 8.4, 2.1 Hz)	5.73-5.99 (1H, *m*)
4	6.12 (1H, *dt*, *J* = 9.9, 3.3 Hz)	5.73-5.99 (1H, *m*)
5	5.95 (1H, *dt*, *J* = 9.9, 1.8 Hz)	6.09 (1H, *ddd*, *J* = 10, 3.5, 1.1 Hz)
6	3.63 (1H, *dd*, *J* = 3.9, 1.8 Hz)	3.62 (1H, *dd*, *J* = 3.5, 2 Hz)
7	4.42 (1H, *d* (AB), *J* = 12 Hz)	4.37 (1H, *d* (AB), *J* = 12 Hz)
	4.68 (1H, *d* (AB), *J* = 12 Hz)	4.70 (1H, *d* (AB), *J* = 12 Hz)
8	-	-
9	-	-
10	-	-
11	2.08 (3H, *s*)	2.06 (3H, *s*)
1' / 1"	-	-
2', 6' / 2", 6"	8.03 (4H, *m*)	7.91-8.12 (4H, *m*)
3',5' / 3",5"	7.45 (4H, *m*)	7.22-7.60 (4H, *m*)
4' / 4"	7.58 (2H, *m*)	7.22-7.60 (2H, *m*)

3.2.2 Compound ST2

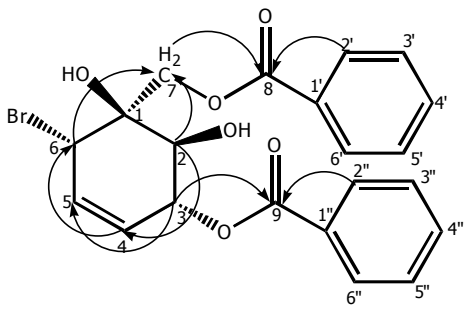


Hydrobromination of compound **SAH1**, pipoxide, with 48% HBr in chloroform under argon atmosphere for 3 hrs gave compound **ST2** in 99.18 % as a white solid, mp. = 198–200 ^{o}C. Its UV absorption spectrum (Fig. 60) showed maxima at 231, 214 and 203 nm. The IR spectrum of compound **ST2** (Fig. 61) showed absorption bands at 3512 (O-H stretching),1694 and 1281 (C=O and C-O stretching) and 709 (C-H bending of monosubstituted phenyl ring). Its molecular formula $C_{21}H_{19}O_6Br$ as indicated by EIMS (Fig. 68) showed the molecular ion peak at 447 *m/z* [M^++1] and 449 [M^++3] in ratio 1:1, indicating a bromine atom in this molecule.

The complete analysis of ^{13}C and ^{1}H NMR spectrum of compound ST2 (see Table 37, Fig. 62 and 63) were assigned with the informations provided from ^{1}H-^{1}H COSY (see Table 35, Fig. 65), HMQC (Fig. 66) and HMBC (see Table 36, Fig. 67) spectra. The ^{13}C NMR spectrum of compound **ST2** recorded in the mixture of DMSO-d_6 in $CDCl_3$ showed 16 signals of 21 carbons. Analysis of the DEPT-135^{o} spectrum (see Table 34, Fig. 64) revealed one methylene carbon (δ 68.8 7), eleven signals of fifteen methine carbons (δ133 .11, 133.04, 129.72 (2xC), 129.64 (2xC), 128.85, 128.35 (2xC), 128.31 (2xC), 126.95, 74.09, 70.05 and 48.94) and four signals of five quaternary carbons (δ1 66.48, 166.43, 130.01 (2xC) and 75.41).

Compound **ST2**, a derivative of compound **SAH1**, showed similar characteristic bands in IR and UV spectrum with compound **SAH1**. Comparison of the ^{1}H NMR spectral data (see Table 38) of the two compounds revealed close structural similarity. Difference in the spectrum of compound **ST2** and **SAH1** was shown as a signal of a methine proton at δ 4.93 (1H-6, *d*, *J* = 4.5 Hz) in **ST2** but absence of the epoxide proton at δ 3.60 (1H-6, *dd*, *J* = 4, 2 Hz) in SAH1, indicating that the epoxide ring was opened. The methylene proton resonating as an AB system at δ 4.84 (1H, *d*, *J* = 12 Hz) and 4.71 (1H, *d*, *J* = 12 Hz) were assigned to H-7a and H-7b, respectively. Two oxymethine protons at δ 5.92 (1H, *br d*, *J* = 8.1 Hz) and 4.40 (1H, *d*, *J* = 8.1 Hz) and two olefinic methine protons at δ 6.07 (1H, *ddd*, *J* = 9.9, 4.5, 1.8 Hz) and 5.78 (1H, *dd*, *J* = 9.9, 2.4 Hz) could be assigned to H-3, H-2, H-5 and H-4, respectively. Ten aromatic protons of the two benzoyl groups showed multiplet signals at δ 8.11 (4H, *m*), 7.64 (2H, *m*) and 7.51 (4H, *m*).

The HMBC correlations of compound **ST2** (see Table 36, Fig.67) were similar to compound **SAH1** except H-7 of ST2 (δ 4.84 and 4.71, 2H, *d* (AB), *J* = 12 Hz) showed correlation peaks with C-8 (166.43), C-1 (75.41), C-2 (70.05) and C-6 (48.94); H-2 (δ 4.40, 1H, *d*, *J* = 8.1 Hz) showed correlation peaks with C-4 (126.95), C-3 (74.09), C-7 (68.87) and C-6 (48.94), thus confirmed the position of bromine atom at C-6 (48.94). The relative stereochemistry shown for compound **ST2** was based on comparison with compound **SAH1**.



Selected HMBC Correlation

By comparison of ^{1}H NMR spectral data with pipoxide bromohydrin (Fongfung, 2001) (see Table 40), compound **ST2** was identified as pipoxide bromohydrin. This compound has been reported before (Fongfung, 2001).

Table 34 ^{13}C and DEPT spectral data of compound **ST2**

δ_C	**DEPT-135°**	**Type of carbon**
166.48		C
166.43		C
133.04	132.73	CH
133.11	132.69	CH
130.01		C
129.72	129.22	CH
129.64	129.20	CH
128.85	128.41	CH
128.35	128.02	CH
128.31	127.98	CH
126.95	126.72	CH
75.41		C
74.09	73.77	CH
70.05	68.94	CH
68.87	68.62	CH_2
48.94	48.92	CH

Table 35 300 MHz COSY correlation of some protons of **ST2**

δ_H (ppm)	Proton correlation with δ_H (ppm)
H-2 (4.40)	H-3 (5.92)
H-4 (5.78)	H-5 (6.07)
H-5 (6.07)	H-4 (5.78), H-6 (4.93)
H-6 (4.93)	H-4 (5.78), H-5 (6.07)
H-7a (4.71)	H-7b (4.84)
H-2', H-6' (8.11)	H-3', H-5' (7.51)
H-2", H-6" (8.11)	H-3", H5" (7.51)
H-3', H-5' (7.51)	H-2', H-6' (8.11), H-4' (7.64)
H-3", H5" (7.51)	H-2", H-6" (8.11), H-4" (7.64)
H-4' (7.64)	H-3', H-5' (7.51)
H-4" (7.64)	H-3", H5" (7.51)

Table 36 Major HMBC correlation of compound **ST2**

Position	δ_H (ppm)	δ_C (ppm)
1	-	-
2	4.40 (1H, *d*, *J* = 8.1 Hz)	C-1 (75.41), C-3 (74.09), C-4 (126.95), C-6 (48.94) and C-7 (68.87)
3	5.92 (1H, *br d*, *J* = 8.1 Hz)	C-2 (70.05), C-4 (126.95), C-5 (128.85) and C-9 (166.48)
4	5.78 (1H, *dd*, *J* = 9.9, 2.4 Hz)	C-2 (70.05), C-5 (128.85) and C-6 (48.94)

Table 36 (Continued)

Position	δ_H (ppm)	δ_C (ppm)
5	6.07 (1H, *ddd*, *J* = 9.9, 4.5, 1.8 Hz)	C-1 (75.41), C-3 (74.06), C-4 (126.95) and C-6 (48.94)
6	4.93 (1H, *d*, *J* = 4.5 Hz)	C-1 (75.41), C-2 (70.05), C-4 (126.95), C-5 (128.85) and C-7 (68.87)
7	4.84 (1H, *d* (AB), *J* = 12 Hz) 4.71 (1H, *d* (AB), *J* = 12 Hz)	C-1 (75.41), C-2 (70.05), C-6 (48.94) and C-8 (166.43)
8	-	-
9	-	-
1'	-	-
2', 6'	8.11 (2H, *m*)	C-8 (166.43) and C-4' [a] (133.11)
3',5'	7.51 (2H, *m*)	C-8 (166.43) and C-1' (130.01)
4'	7.64 (1H, *m*)	-
1"	-	-
2", 6"	8.11 (2H, *m*)	C-9 (166.48) and C-4" [a] (133.04)
3",5"	7.51 (2H, *m*)	C-9 (166.48) and C-1" (130.01)
4"	7.64 (1H, *m*)	-

[a] May be interchangeable.

Table 37 ^{1}H and ^{13}C NMR spectral data of compound **ST2**

Position	δ_C [#] (ppm)		δ_H (ppm)
1	75.41	C	-
2	70.05	CH	4.40 (1H, *d*, *J* = 8.1 Hz)
3	74.09	CH	5.92 (1H, *br d*, *J* = 8.1 Hz)
4	126.95	CH	5.78 (1H, *dd*, *J* = 9.9, 2.4 Hz)
5	128.85	CH	6.07 (1H, *ddd*, *J* = 9.9, 4.5, 1.8 Hz)
6	48.94	CH	4.93 (1H, *d*, *J* = 4.5 Hz)
7	68.87	CH_2	4.84 (1H, *d* (AB), *J* = 12 Hz)
			4.71 (1H, *d* (AB), *J* = 12 Hz)
8	166.43	C	-
9	166.48	C	-
1'	130.01	C	-
2', 6' [a]	129.72	CH	8.11 (2H, *m*)
3',5' [b]	128.35	CH	7.51 (2H, *m*)
4' [c]	133.11	CH	7.64 (1H, *m*)
1"	130.01	C	-
2", 6" [a]	129.64	CH	8.11 (2H, *m*)
3",5" [b]	128.31	CH	7.51 (2H, *m*)
4" [c]	133.04	CH	7.64 (1H, *m*)

[#] Carbon type deduced from DEPT experiment.

[a, b, c] May be interchangeable.

Table 38 Comparison of ^{1}H NMR spectral data between compound **ST2** and **SAH1**

Position	**Compound ST2, δ_H (ppm), Dissolved in $CDCl_3$ + DMSO-d_6**	**Compound SAH1, δ_H (ppm), Dissolved in $CDCl_3$**
1	-	-
2	4.40 (1H, *d*, *J* = 8.1 Hz)	4.33 (1H, *dd*, *J* = 8, 6 Hz)
3	5.92 (1H, *br d*, *J* = 8.1 Hz)	5.67 (1H, *ddd*, *J* = 8, 3, 2 Hz)
4	5.78 (1H, *dd*, *J* = 9.9, 2.4 Hz)	5.91 (1H, *dt*, *J* = 10, 2 Hz)
5	6.07 (1H, *ddd*, *J* = 9.9, 4.5, 1.8 Hz)	6.10 (1H, *ddd*, *J* = 10, 4, 2 Hz)
6	4.93 (1H, *d*, *J* = 4.5 Hz)	3.60 (1H, *dd*, *J* = 4, 2 Hz)
7	4.84 (1H, *d* (AB), *J* = 12 Hz) 4.71 (1H, *d* (AB), *J* = 12 Hz)	4.48 (1H, *d* (AB), *J* = 12 Hz) , 5.00 (1H, *d* (AB), *J* = 12 Hz)
8	-	-
9	-	-
1'	-	-
2', 6'	8.11 (2H, *m*)	8.06 (2H, *m*)
3',5'	7.51 (2H, *m*)	7.45 (2H, *m*)
4'	7.64 (1H, *m*)	7.58 (1H, *m*)
1"	-	-
2", 6"	8.11 (2H, *m*)	8.06 (2H, *m*)
3",5"	7.51 (2H, *m*)	7.45 (2H, *m*)
4"	7.64 (1H, *m*)	7.58 (1H, *m*)

Table 39 Comparison of ^{13}C NMR spectral data between compound **ST2** and **SAH1**

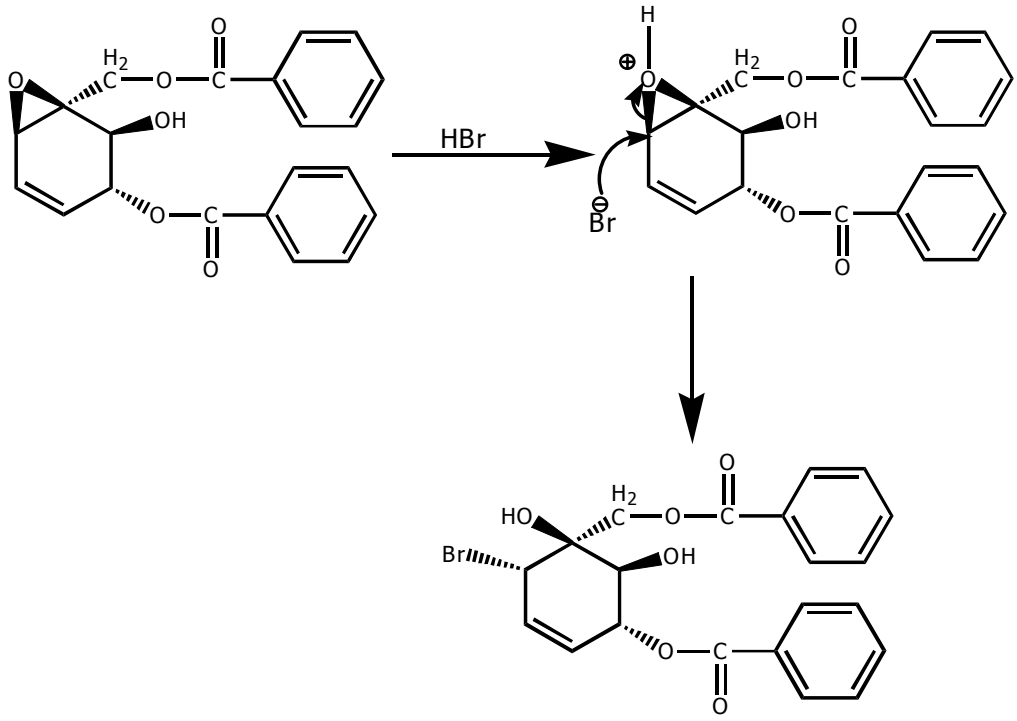
Position	ST2, δ_C (ppm), Dissolved in $CDCl_3$ + DMSO-d_6	SAH1, δ_C (ppm), Dissolved in $CDCl_3$
1	75.41	59.49
2	70.05	71.06
3	74.09	74.82
4	126.95	132.96
5	128.85	124.73
6	48.94	54.20
7	68.87	62.91
8	166.43	166.20
9	166.48	166.87
1' [a]	130.01	129.40
2', 6' [b]	129.72	129.78
3',5' [c]	128.35	128.43
4' [d]	133.11	133.38
1" [a]	130.01	129.44
2", 6" [b]	129.64	129.84
3",5" [c]	128.31	128.47
4" [d]	133.04	133.44

[a, b, c, d] May be interchangeable.

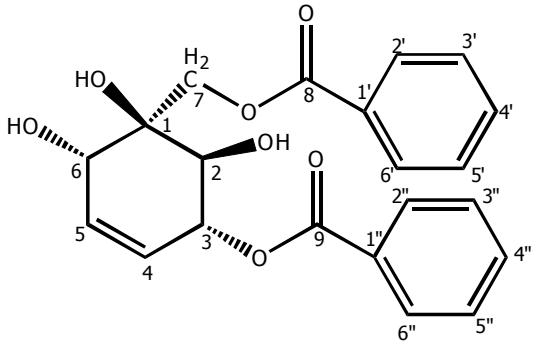
Table 40 Comparison of ^{1}H NMR spectral data between compound **ST2** and **pipoxide bromohydrin**

Position	ST2, δ_H (ppm), Dissolved in $CDCl_3$ + DMSO-d_6	Pipoxide bromohydrin, δ_H (ppm), Dissolved in $CDCl_3$ + DMSO-d_6
1	-	-
2	4.40 (1H, *d*, *J* = 8.1 Hz)	4.31 (1H, *br t*, *J* = 7 Hz)
3	5.92 (1H, *br d*, *J* = 8.1 Hz)	5.84 (1H, *br d*, *J* = 8 Hz)
4	5.78 (1H, *dd*, *J* = 10.2, 2.4 Hz)	5.65 (1H, *dd*, *J* = 10, 3 Hz)
5	6.07 (1H, *ddd*, *J* = 9.9, 4.5, 1.8 Hz)	5.95 (1H, *ddd*, *J* = 10, 5, 2 Hz)
6	4.93 (1H, *d*, *J* = 4.5 Hz)	4.73 (1H, *d*, *J* = 5 Hz)
7	4.84 (1H, *d* (AB), *J* = 12 Hz)	4.85 (1H, *d* (AB), *J* = 12 Hz)
	4.71 (1H, *d* (AB), *J* = 12 Hz)	4.63 (1H, *d* (AB), *J* = 12 Hz)
8	-	-
9	-	-
1'	-	-
2', 6'	8.11 (2H, *m*)	7.98 (2H, *m*)
3',5'	7.51 (2H, *m*)	7.34 (2H, *m*)
4'	7.64 (2H, *m*)	7.47 (1H, *m*)
1"	-	-
2", 6"	8.11 (2H, *m*)	7.98 (2H, *m*)
3",5"	7.51 (2H, *m*)	7.34 (2H, *m*)
4"	7.64 (2H, *m*)	7.47 (1H, m)

Scheme 13 Mechanism for the hydrobromination of compound ST2



3.2.2 Compound ST3



Compound **ST3** was obtained from hydrolysis of compound **SAH1** with 4N H_2SO_4 in dioxane at room temperature for 22 hrs. under nitrogen atmosphere, as a

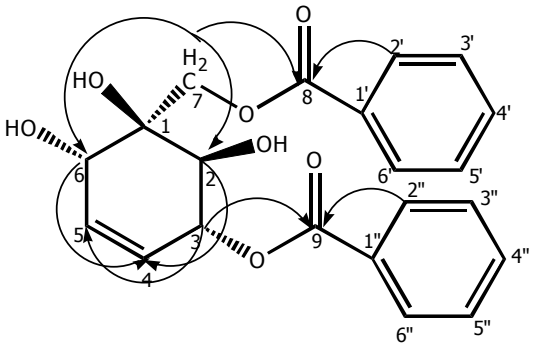
colorless viscous liquid, $[\alpha]_D^{26.6}$ = - 78.95° (c = 0.038, $CHCl_3$). Its UV absorption spectrum (Fig. 69) showed maxima at 230, 212 and 202 nm. The IR spectrum of compound **ST3** (Fig. 70) showed absorption bands at 3438 (OH stretching), 1704 (C=O stretching), 1278 (C-O stretching) and 709 cm^{-1}(C-H bending of monosubstitued phenyl ring).

The complete analysis of ^{13}C and ^{1}H NMR spectral data of compound **ST3** (see Table 44, Fig. 71 and 72) were assigned with informations provided from ^{1}H-^{1}H COSY (see Table 42, Fig. 74), ^{1}H-^{13}C correlation (HMQC) (Fig. 75) and ^{1}H-^{13}C correlation by long-range coupling (HMBC) (see Table 43 , Fig. 76), along with comparison of ^{1}H NMR spectral data with compound **SAH1** (see Table 45). The ^{13}C NMR spectrum of compound **ST3** showed 13 signals for 21 carbons. The DEPT-135° (see Table 41, Fig. 73) indicated the existence of one methylene carbon (δ 66.76), eight signals of fifteen methine carbons (δ 13 3.48 (2xC), 129.82 (4xC), 129.54, 128.45 (4xC), 126.87, 74.30, 70.86 and 68.56) and four signals of five quaternary carbons (δ 167.82, 167.15, 133.41 (2xC) and 75.92). The mass spectrum of this compound (Fig. 79) showed the molecular ion peak at 407 *m/z* ($[M^+$+Na]), thus this compound had molecular formula $C_{21}H_{20}O_7$.

Compound **ST3**, a derivative of compound **SAH1**, showed similar characteristic bands in IR and UV spectrum with **SAH1**. Comparison of the ^{1}H NMR spectral data (see Table 45) of the two compounds revealed close structural similarity. Difference in the spectrum of compound **ST3** was shown as a signal of oxymethine proton at δ 4.33 (1H, *br d*, *J* = 3.9 Hz) which was shown at the lower field than epoxy methine proton (δ 3 .60, 1H, *dd*, *J* = 4, 2 Hz) in compound **SAH1**, indicating that epoxide ring was opened. Two olefinic methine protons appearing at δ 6.02 (1H, *ddd*, *J* = 10.2, 3.9, 1.5 Hz) and 5.88 (1H, *ddd*, *J* = 10.2, 3, 1 Hz) could be assigned to H-5 and H-4, respectively. The oxymethine protons at C-2 and C-3 were resonated at δ 4.24 (1H, *d*, *J* = 6 Hz) and 5.70 (1H, *m*), respectively. Doublet AB system at δ

4.89 (1H, *d*, *J* = 12.3 Hz) and 4.75 (1H, *d*, *J* = 12.3 Hz) were assigned to H-7b and H-7a, respectively. In addition, ^{1}H NMR spectrum of two benzoyl groups showed signals at δ 8.03 (2H, *m*), 7.98 (2H, *m*), 7.56 (2H, *m*) and 7.40 (4H, *m*).

The HMBC correlation of compound **ST3** (see Table 43, Fig. 76) showed the same correlation with compound **SAH1**. From NOE experiment (Fig. 77 and 78), irradiation of H-7b (δ 4.89) showed enhancement of H-7a (δ 4.75) and H-2 (δ 4.24). Irradiation of H-7a (δ 4.75) showed enhancement of H-7b (δ 4.89). Irradiation of H-6 (δ 4.33) showed enhancement of H-5 (δ 6.02). Irradiation of H-3 (δ 5.70) showed enhancement of H-4 (δ 5.88). Irradiation of H-2 (δ 4.24) showed enhancement of H-7b (δ 4.89) and H-7a (δ 4.75). Thus, this result suggested that the two hydroxyl groups at position 1 and 2 were on the same side and opposite side to the substituents at the position 3 and 6.



Selected HMBC Correlation

By comparison of ^{1}H and ^{13}C NMR spectral data between compound **ST3** and **SAH3** previously isolated from the leaves of *U. purpurea* Blume in this work (see Table 46 and 47); between compound **ST3** and **(-)-zeylenol** (see Table 48 and 49) which was the compound previously isolated from the roots of *Uvaria zeylanica* L. (Jolad, *et al*., 1981) (see Table 48, 49), all compounds showed similarity. The relative stereochemistry of compound **ST3** was deduced from optical rotation $[\alpha]_D^{26.6}$

of - 78.95° (c = 0.038 g/100 cm^{-3}, $CHCl_3$) which was identical to that of compound **SAH3** value, $[\alpha]_D^{25.9}$ of - 90.90° (c = 0.011, $CHCl_3$) and (-)-**zeylenol** value with $[\alpha]_D$ of - 116.3° (c = 0.915, $CHCl_3$) (Jolad, *et al*., 1981) . Thus compound **ST3** was identified as **zeylenol**.

Table 41 ^{13}C and DEPT spectral data of compound **ST3**

δ_C	**DEPT-135°**	**Type of Carbon**
167.82		C
167.15		C
133.48	133.51	CH
133.41		C
129.82	129.84	CH
129.54	129.53	CH
128.45	128.47	CH
126.87	126.88	CH
75.92		C
74.30	74.31	CH
70.86	70.86	CH
68.56	68.55	CH
66.76	66.76	CH_2

Table 42 300 MHz COSY correlation of some protons of **ST3**

δ_H (ppm)	Proton correlation with δ_H (ppm)
H-2 (4.24)	H-3 (5.70)
H-3 (5.70)	H-2 (4.24), H-4 (5.88)
H-4 (5.88)	H-3 (5.70), H-5 (6.02)
H-5 (6.02)	H-4 (5.88), H-6 (4.33)
H-6 (4.33)	H-5 (6.0)
H-7a (4.75)	H-7b (4.89)
H-2', H-6', (7.98)	H-3', H-5' (7.40)
H-2", H-6"(8.03)	H-3", H5" (7.40)
H-3', H-5' (7.40)	H-2', H-6' (7.98), H-4' (7.56)
H-3", H5" (7.40)	H-2", H-6" (8.03), H-4" (7.56)
H-4' (7.56)	H-3', H-5' (7.40)
H-4" (7.56)	H-3", H5" (7.40)

Table 43 Major HMBC correlation of compound **ST3**

Position	δ_H (ppm)	δ_C (ppm)
1	-	-
2	4.24 (1H, *d*, *J* = 6 Hz)	C-3 (74.30) and C-4 (126.87)
3	5.70 (1H, *m*)	C-2 (70.86), C-4 (126.87), C-5 (129.54) and C-9 (167.15)
4	5.88 (1H, *ddd*, *J* = 10.2, 3, 1 Hz)	C-2 (70.86)
5	6.02 (1H, *ddd*, *J* = 10.2, 3.9, 1.5 Hz)	-

Table 43 (Continued)

Position	δ_H (ppm)	δ_C (ppm)
6	4.33 (1H, *br d*, *J* = 3.9 Hz)	C-1 (75.92), C-4 (126.87) and C-5 (129.54)
7	**4.75 (1H, *d* (AB), *J* = 12.3 Hz)** **4.89 (1H, *d* (AB), *J* = 12.3 Hz)**	C-1 (75.92), C-2 (70.86), C-6 (68.56) and C-8 (167.82)
8	-	-
9	-	-
1'	-	-
2', 6'	7.98 (2H, *m*)	C-1' (133.41), C-3', C-5' (128.45), C-4' (133.48) and C-8 (167.82)
3',5'	7.40 (2H, *m*)	C-2',C-6' (129.82)
4'	7.56 (1H, *m*)	C-2',C-6' (129.82)
1"	-	-
2", 6"	8.03 (2H, *m*)	C-1" (133.41), C-3", C-5" (128.45), C-4" (133.48) and C-9 (167.15)
3",5"	7.40 (2H, *m*)	C-2",C-6" (129.82)
4"	7.56 (1H, *m*)	C-2",C-6" (129.82)

Table 44 ^{1}H and ^{13}C NMR spectral data of compound **ST3**

Position	δ_C [#] (ppm)		δ_H (ppm)
1	75.92	C	-
2	70.86	CH	4.24 (1H, *d*, *J* = 6 Hz)
3	74.30	CH	5.70 (1H, *m*)
4	126.87	CH	5.88 (1H, *ddd*, *J* = 10.2, 3, 1 Hz)
5	129.54	CH	6.02 (1H, *ddd*, *J* = 10.2, 3.9, 1.5 Hz)
6	68.56	CH	4.33 (1H, *br d*, *J* = 3.9 Hz)
7	66.76	CH_2	**4.75 (1H, *d* (AB), *J* = 12.3 Hz)**
			4.89 (1H, *d* (AB), *J* = 12.3 Hz)
8	167.82	C	-
9	167.15	C	-
1'	133.41	C	-
2', 6'	129.82	CH	7.98 (2H, *m*)
3',5'	128.45	CH	7.40 (2H, *m*)
4'	133.48	CH	7.56 (1H, *m*)
1"	133.41	C	-
2", 6"	129.82	CH	8.03 (2H, *m*)
3",5"	128.45	CH	7.40 (2H, *m*)
4"	133.48	CH	7.56 (1H, *m*)

[#] Carbon type deduced from DEPT experiment.

Table 45 Comparison of ^{1}H NMR spectral data between compound **ST3** and **SAH1**

Position	Compound ST3, δ_H (ppm)	Compound SAH1, δ_H (ppm)
1	-	-
2	4.24 (1H, *d*, *J* = 6 Hz)	4.33 (1H, *dd*, *J* = 8, 6 Hz)
3	5.70 (1H, *m*)	5.67 (1H, *ddd*, *J* = 8, 3, 2 Hz)
4	5.88 (1H, *ddd*, *J* = 10.2, 3, 1 Hz)	5.91 (1H, *dt*, *J* = 10, 2 Hz)
5	6.02 (1H, *ddd*, *J* = 10.2, 3.9, 1.5 Hz)	6.10 (1H, *ddd*, *J* = 10, 4, 2 Hz)
6	4.33 (1H, *br d*, *J* = 3.9 Hz)	3.60 (1H, *dd*, *J* = 4, 2 Hz)
7	**4.75 (1H, *d* (AB), *J* = 12.3 Hz)**	4.48(1H, *d* (AB), *J* = 12 Hz)
	4.89 (1H, *d* (AB), *J* = 12.3 Hz)	5.00 (1H, *d* (AB), *J* = 12 Hz)
8	-	-
9	-	-
1'	-	-
2', 6'	7.98 (2H, *m*)	8.06 (2H, *m*)
3',5'	7.40 (2H, *m*)	7.45 (2H, *m*)
4'	7.56 (1H, *m*)	7.58 (1H, *m*)
1"	-	-
2", 6"	8.03 (2H, *m*)	8.06 (2H, *m*)
3",5"	7.40 (2H, *m*)	7.45 (2H, *m*)
4"	7.56 (1H, *m*)	7.58 (1H, *m*)

Table 46 Comparison of ^{1}H NMR spectral data between compound **ST3** and **SAH3**

Position	Compound ST3, δ_H (ppm)	Compound SAH3, δ_H (ppm)
1	-	-
2	4.24 (1H, *d*, *J* = 6 Hz)	4.24 (1H, *d*, *J* = 6 Hz)
3	5.70 (1H, *m*)	5.70 (1H, *m*)
4	5.88 (1H, *ddd*, *J* = 10.2, 3, 1 Hz)	5.84 (1H, *ddd*, *J* = 10, 2.5, 1 Hz)
5	6.02 (1H, *ddd*, *J* = 10.2, 3.9, 1.5 Hz)	5.99 (1H, *ddd*, *J* = 10, 4, 1.5 Hz)
6	4.33 (1H, *br d*, *J* = 3.9 Hz)	4.34 (1H, *d*, *J* = 4 Hz)
7	**4.75 (1H, *d* (AB), *J* = 12.3 Hz)**	4.72 (1H, *d* (AB), *J* = 12 Hz)
	4.89 (1H, *d* (AB), *J* = 12.3 Hz)	4.87 (1H, *d* (AB), *J* = 12 Hz)
8	-	-
9	-	-
1'	-	-
2', 6'	7.98 (2H, *m*)	7.94 (2H, *m*)
3',5'	7.40 (2H, *m*)	7.37 (2H, *m*)
4'	7.56 (1H, *m*)	7.52 (1H, *m*)
1"	-	-
2", 6"	8.03 (2H, *m*)	7.99 (2H, *m*)
3",5"	7.40 (2H, *m*)	7.37 (2H, *m*)
4"	7.56 (1H, *m*)	7.52(1H, *m*)

Table 47 Comparison of ^{13}C NMR spectral data between compound **ST3** and **SAH3**

Position	ST3, δ_C (ppm)	SAH3, δ_C (ppm)
1	75.92	75.90
2	70.86	70.79
3	74.30	74.10
4	126.87	126.71
5	129.54	129.76
6	68.56	68.68
7	66.76	66.67
8	167.82	167.78
9	167.15	167.09
1'	133.41	129.23
2', 6'	129.82	129.78
3',5'	128.45	128.41
4'	133.48	133.42
1"	133.41	129.43
2", 6"	129.82	129.81
3",5"	128.45	128.38
4"	133.48	133.37

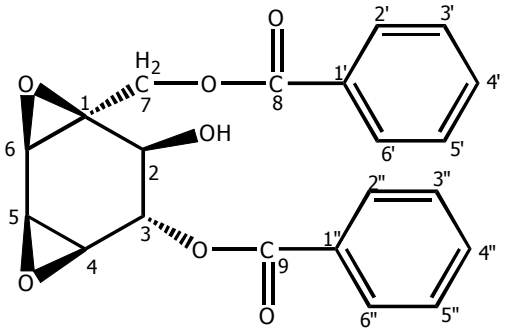
Table 48 Comparison of ^{1}H NMR spectral data between compound **ST3** and (-)-zeylenol

Position	ST3, δ_H (ppm)	(-)-Zeylenol, δ_H (ppm)
1	-	-
2	4.24 (1H, *d*, *J* = 6 Hz)	4.22 (1H, *d*, *J* = 6.1 Hz)
3	5.70 (1H, *m*)	5.70 (1H, *dddd*, *J* = 6.1, 2.6, 1.6, 1.1 Hz)
4	5.88 (1H, *ddd*, *J* = 10.2, 3, 1 Hz)	5.88 (1H, *ddd*, *J* = 10.1, 2.6, 0.7 Hz)
5	6.02 (1H, *ddd*, *J* = 10.2, 3.9, 1.5 Hz)	5.99 (1H, *ddd, J* = 10.1, 4, 1.6 Hz)
6	4.33 (1H, *br d*, *J* = 3.9 Hz)	4.32 (1H, *ddd*, *J* = 4, 1.1, 0.7 Hz)
7	**4.75 (1H, *d* (AB), *J* = 12.3 Hz)**	4.75 (1H, *d* (AB), *J* = 12.3 Hz) 4.89 (1H, *d* (AB), *J* = 12.3 Hz)
8	4.89 (1H, *d* (AB), *J* = 12.3 Hz)	-
9	-	-
1',1"	-	-
2', 6' / 2", 6"	-	7.89 (2H, *m*) / 8.02 (2H, *m*)
3', 5' / 3", 5"	7.98 (2H, *m*) / 8.03 (2H, *m*)	7.40 (4H, *m*)
4' / 4"	7.40 (4H, *m*) 7.56 (2H, *m*)	7.55 (2H, *m*)

Table 49 Comparison of ^{13}C NMR spectral data between compound **ST3** and **(-)-zeylenol**

Position	ST3, δ_C (ppm)	(-)-Zeylenol, δ_C (ppm)
1	75.92	76.0
2	70.86	68.7
3	74.30	74.4
4	126.87	127.0
5	129.54	129.5
6	68.56	70.9
7	66.76	66.8
8	167.82	165.0
9	167.15	165.0
1'	133.41	128.5
2', 6'	129.82	129.9
3', 5'	128.45	128.5
4'	133.48	133.5
1"	133.41	128.5
2", 6"	129.82	129.9
3", 5"	128.45	128.5
4"	133.48	133.5

3.2.4 Compound ST4



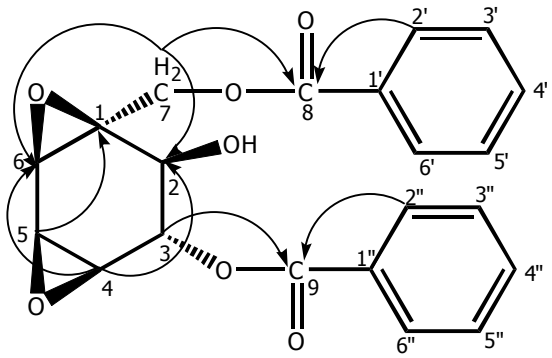
Compound **ST4** was obtained from epoxidation of compound **SAH1** (pipoxide) with *m*-CPBA in methylene chloride at room temperature for 23 hrs under nitrogen atmosphere as a colorless viscous liquid, $[\alpha]_D^{27.0}$ = - 61.2° (*c* = 0.049, $CHCl_3$). It UV absorption spectrum (Fig. 80) showed maxima at 274, 229 and 203 nm. The IR spectrum of compound **ST4** (Fig. 81) showed absorption bands at 3482 (O-H stretching), 1719 (C=O stretching), 1272 and 709 cm^{-1} (C=C stretching and C-H bending of monosubstituted phenyl ring).

The ^{13}C NMR spectrum of compound **ST4** (see Table 50, Fig. 83) recording in $CDCl_3$ showed 16 signals for 21 carbon atoms. The DEPT-90° and DEPT-135° (see Table 50, Fig. 84) indicated the existence of one methylene carbon atom (δ 62.55), eleven signals of fifteen methine carbon atoms (δ 133.56 (2xC), 133.49, 129.99 (2xC), 129.84 (2xC), 128.52, 128.48 (2xC), 73.82, 68.01, 57.40, 53.73 and 52.06) and four signals of five quaternary carbon atoms (δ 166.74, 166.20, 129.30 and 61.10).

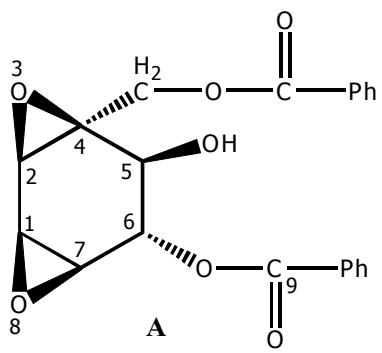
Compound **ST4**, a derivative of compound **SAH1**, showed the same characteristic peaks in the IR and UV spectrum with compound **SAH1**. Comparison of ^1H NMR spectrum (see Table 54) of the two compounds revealed their close structural similarity. Difference in the spectrum of compound **ST4** was shown in the disappearance of two olefinic methine protons at δ 5.91 (1H, *dt*, *J* = 10, 2 Hz) and

6.10 (1H, *ddd*, *J* = 10, 4, 2 Hz) which was observed in compound **SAH1**. ^{1}H NMR spectrum of compound **ST4** showed five oxymethine protons at δ 5.51 (1H, *dd*, *J* = 8.1, 0.6 Hz), 4.42 (1H, *d*, *J* = 8.1 Hz), 3.78 (1H, *d*, *J* = 2.4 Hz), 3.65 (1H, *dd*, *J* = 4.5, 2.4 Hz) and 3.50 (1H, *br d*, *J* = 4.5 Hz) which were assigned to H-3, H-2, H-6, H-5 and H-4, respectively. Signals of ten aromatic protons appeared at δ 8.08 (4H, *m*), 7,58 (2H, *m*) and 7.45 (4H, *m*). The doublet AB system of methylene proton resonated at δ 4.91 (1H, *d*, *J* = 12 Hz) and 4.32 (1H, *d*, *J* = 12 Hz).

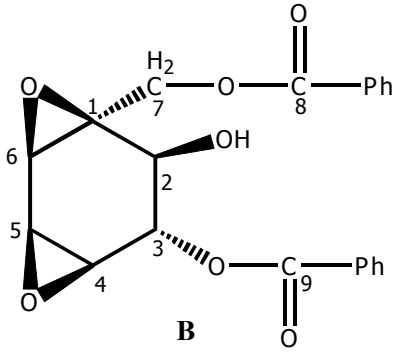
The structure of compound **ST4** was deduced from its ^{13}C and ^{1}H NMR spectral data (see Table 53, Fig. 82 and 83) together with the results of ^{1}H-^{1}H COSY (Fig. 85), HMQC (Fig. 86) and HMBC (see Table 52, Fig. 87). The correlation peaks in the HMBC spectrum of H-6 (δ 3.78) with the carbons at δ 62.55 (C-7), 61.10 (C-1), 53.73 (C-4) and 52.06 (C-5); of H-4 (δ 3.50) with the carbons at δ 73.82 (C-3), 68.01 (C-2), 57.40 (C-6) and 52.06 (C-5), confirmed that the epoxide ring was formed between C-4 (δ 53.73) and C-5 (δ 52.06). From NOE experiment (Fig. 88 and 89), irradiation of H-7b (δ 4.91) resulted in the enhancement of the signals at H-7a (δ 4.32) and H-2 (δ 4.42). Irradiation of H-7a (δ 4.32) resulted in the enhancement of the signals at H-7b (δ 4.91) and H-6 (δ 3.78). Irradiation of H-6 (δ 3.78) resulted in the enhancement of the signals at H-7a (δ 4.32) and H-5 (δ 3.65). Irradiation of H-5 (δ 3.65) resulted in the enhancement of the signals at H-6 (δ 3.78), H-4 (δ 3.50) and H-3 (δ 5.51). Irradiation of H-4 (δ 3.50) resulted in the enhancement of the signals at H-5 (δ 3.65) and H-3 (δ 5.51). Irradiation of H-3 (δ 5.51) resulted in the enhancement of signal at H-4 (δ 3.50). No enhancement was observed on H-2 (δ 4.42). Irradiation of H-2 (δ 4.42) resulted in the enhancement of the signal at H-7b (4.91) but not the signal at H-3 (δ 5.51). Thus, this result suggested that the two epoxide ring were on the same side and opposite side to the benzoyl group at the position 3.



Selected HMBC Correlation



A



B

* Systematic number is shown in **A**; to retain consistency with the thesis, the conventional cyclohexane numbering shown in **B** is used throughout.

Comparison of ^{1}H and ^{13}C NMR spectra and specific rotation between compound **ST4** and **diepoxide**, (-)-(1R,2R,4R,5S,6R,7R)-4-Benzoyloxymethyl-3,8-dioxatricyclo [5,1,0,$0^{2,4}$] -octane-5,6-diol 6-benzoate* (Pancharoen, *et al*., 1996) (see Table 57, 56) showed similarity with the optical rotation $[\alpha]_D^{27.0}$ of - 61.2^{o} (*c* = 0.049, $CHCl_3$) being identical to the reported value, $[\alpha]_D^{22}$ of - 76^{o} (*c* = 0.16, $CHCl_3$). Thus, compound **ST4** was identified as (-)-(1R,2R,4R,5S,6R,7R)-4-Benzoyloxymethyl-3,8-dioxatricyclo [5,1,0,$0^{2,4}$] -octane-5,6-diol 6-benzoate.

Table 50 ^{13}C and DEPT spectral data of compound **ST4**

δ_C	**DEPT-90°**	**DEPT-135°**	**Type of Carbon**
166.74			C
166.20			C
133.56	133.57	133.57	CH
133.49	133.50	133.50	CH
129.99	130.00	129.99	CH
129.84	129.85	129.84	CH
129.30			C
128.52	128.54	128.53	CH
128.48	128.48	128.48	CH
73.82	73.82	73.80	CH
68.01	68.01	68.00	CH
62.55		62.55	CH_2
61.10			C
57.40	57.41	57.40	CH
53.73	53.75	53.75	CH
52.06	52.08	52.07	CH

Table 51 300 MHz COSY correlation of some protons of **ST4**

δ_H (ppm)	Proton correlation with δ_H (ppm)
H-2 (4.42)	H-3 (5.51)
H-3 (5.51)	H-2 (4.42), H-4 (3.50)
H-4 (3.50)	H-3 (5.51), H-5 (3.65)
H-5 (3.65)	H-4 (3.50), H-6 (3.78)
H-6 (3.78)	H-5 (3.65)
H-7a (4.32)	H-7b (4.91)
H-2', H-6', (8.08)	H-3', H-5' (7.45), H-4' (7.58)
H-2", H-6"(8.08)	H-3", H5" (7.45), H-4" (7.58)
H-3', H-5' (7.45)	H-2', H-6' (8.08), H-4' (7.58)
H-3", H5" (7.45)	H-2", H-6" (8.08), H-4" (7.58)
H-4' (7.58)	H-2', H-6' (8.08), H-3', H-5' (7.45)
H-4" (7.58)	H-2", H-6" (8.08), H-3", H5" (7.45)

Table 52 Major HMBC correlation of compound **ST4**

Position	δ_H (ppm)	δ_C (ppm)
1	-	-
2	4.42 (1H, *d*, *J* = 8.1 Hz)	C-3 (73.82)
3	5.51 (1H, *dd*, *J* = 8.1, 0.6 Hz)	C-2 (68.01) and C-9 (166.74)
4	3.50 (1H, *br d*, *J* = 4.5 Hz)	C-2 (68.01), C-3 (73.82), C-5 (52.06) and C-6 (57.40)
5	3.65 (1H, *dd*, *J* = 4.5, 2.4 Hz)	C-1 (61.10) and C-6 (57.40)
6	3.78 (1H, *d*, *J* = 2.4 Hz)	C-1 (61.10), C-4 (53.73), C-5 (52.06) and C-7 (62.55)

Table 52 (Continued)

Position	δ_H (ppm)	δ_C (ppm)
7	**4.32 (1H, *d* (AB), *J* = 12 Hz)**	C-1 (61.10), C-2 (68.01), C-6
	4.91 (1H, *d* (AB), *J* = 12 Hz)	(57.40) and C-8 (166.20)
8	-	-
9	-	-
1'	-	-
2', 6'	8.08 (2H, *m*)	C-1' (129.30) and C-8 (166.20)
3', 5'	7.45 (2H, *m*)	C-1' (129.30) and C-8 (166.20)
4'	7.58 (1H, *m*)	C-1' (129.30) and C-2', C-6'
		(129.99)
1"	-	-
2", 6"	8.08 (2H, *m*)	C-3", C-5" (133.56), C-4"
		(133.49) and C-9 (166.74)
3", 5"	7.45 (2H, *m*)	C-1" (129.30) and C-9 (166.74)
4"	7.58 (1H, *m*)	C-2",C-6" (129.84) and C-1"
		(129.30)

Table 53 ^{1}H and ^{13}C NMR spectral data of compound **ST4**

Position	δ_C [#] (ppm)		δ_H (ppm)
1	61.10	C	-
2	68.01	CH	4.42 (1H, *d*, *J* = 8.1 Hz)
3	73.82	CH	5.51 (1H, *dd*, *J* = 8.1, 0.6 Hz)
4	53.73	CH	3.50 (1H, *br d*, *J* = 4.5 Hz)
5	52.06	CH	3.65 (1H, *dd*, *J* = 4.5, 2.4 Hz)
6	57.40	CH	3.78 (1H, *d*, *J* = 2.4 Hz)
7	62.55	CH_2	**4.32 (1H, *d* (AB), *J* = 12 Hz)**
			4.91 (1H, *d* (AB), *J* = 12 Hz)
8	166.20	C	-
9	166.74	C	-
1'	129.30	C	-
2', 6' [a]	129.99	CH	8.08 (2H, *m*)
3', 5' [b]	128.48	CH	7.45 (2H, *m*)
4' [c]	128.52	CH	7.58 (1H, *m*)
1"	129.30	C	-
2", 6" [a]	129.84	CH	8.08 (2H, *m*)
3", 5" [b]	133.56	CH	7.45 (2H, *m*)
4" [c]	133.49	CH	7.58 (1H, *m*)

[#] Carbon type deduced from DEPT experiment.

[a, b, c] May be interchangeable.

Table 54 **Comparison of ^{1}H NMR spectral data between** ST4 **and** SAH1

Position	**Compound ST4, δ_H (ppm)**	**Compound SAH1, δ_H (ppm)**
1	-	-
2	4.42 (1H, *d*, *J* = 8.1 Hz)	4.33 (1H, *dd*, *J* = 8, 6 Hz)
3	5.51 (1H, *dd*, *J* = 8.1, 0.6 Hz)	5.67 (1H, *ddd*, *J* = 8, 3, 2 Hz)
4	3.50 (1H, *br d*, *J* = 4.5 Hz)	5.91 (1H, *dt*, *J* = 10, 2 Hz)
5	3.65 (1H, *dd*, *J* = 4.5, 2.4 Hz)	6.10 (1H, *ddd*, *J* = 10, 4, 2 Hz)
6	3.78 (1H, *d*, *J* = 2.4 Hz)	3.60 (1H, *dd*, *J* = 4, 2 Hz)
7	**4.32 (1H, *d* (AB), *J* = 12 Hz)**	4.48(1H, *d* (AB), *J* = 12 Hz)
	4.91 (1H, *d* (AB), *J* = 12 Hz)	5.00 (1H, *d* (AB), *J* = 12 Hz)
8	-	-
9	-	-
1'	-	-
2', 6'	8.08 (2H, *m*)	8.06 (2H, *m*)
3',5'	7.45 (2H, *m*)	7.45 (2H, *m*)
4'	7.58 (1H, *m*)	7.58 (1H, *m*)
1"	-	-
2", 6"	8.08 (2H, *m*)	8.06 (2H, *m*)
3",5"	7.45 (2H, *m*)	7.45 (2H, *m*)
4"	7.58 (1H, *m*)	7.58 (1H, *m*)

Table 55 **Comparison of ^{13}C NMR spectral data between** ST4 **and** SAH1

Position	**Compound ST4, δ_C (ppm)**	**Compound SAH1, δ_C (ppm)**
1	61.10	59.49
2	68.01	71.06
3	73.82	74.82
4	53.73	132.96
5	52.06	124.73
6	57.40	54.20
7	62.55	62.91
8	166.20	166.20
9	166.74	166.87
1'	129.30	129.40
2', 6' [a]	129.99	129.78
3',5' [b]	128.48	128.43
4' [c]	128.52	133.38
1"	129.30	129.44
2", 6" [a]	129.84	129.84
3",5" [b]	133.56	128.47
4" [c]	133.49	133.44

[a, b, c] May be interchangeable.

Table 56 **Comparison of ^{1}H NMR spectral data between compound** ST4 **and** diepoxide

Position	**Compound ST4, δ_H (ppm)**	**Diepoxide, δ_H (ppm)**
1	-	-
2	4.42 (1H, *d*, *J* = 8.1 Hz)	4.30 (1H, *dd*, *J* = 10, 5.1 Hz)
3	5.51 (1H, *dd*, *J* = 8.1, 0.6 Hz)	5.44(1H, *dd*, *J* = 5.1, 2.5 Hz)
4	3.50 (1H, *br d*, *J* = 4.5 Hz)	3.46 (1H, *ddd*, *J* = 3.5, 2.5, 0.75 Hz)
5	3.65 (1H, *dd*, *J* = 4.5, 2.4 Hz)	3.60 (1H, *dd*, *J* = 3.5, 2.75 Hz)
6	3.78 (1H, *d*, *J* = 2.4 Hz)	3.74 (1H, *d*, *J* = 2.75 Hz)
7	**4.32 (1H, *d* (AB), *J* = 12 Hz)**	4.49 (1H, *d*, *J* = 12 Hz)
		4.61 (1H, *d*, *J* = 12 Hz)
8	**4.91 (1H, *d* (AB), *J* = 12 Hz)**	-
9		-
1'	-	-
2', 6'	-	7.99 (2H, *m*)
3',5'	-	7.38 (2H, *m*)
4'	8.08 (2H, *m*)	7.55 (1H, *m*)
1"	7.45 (2H, *m*)	-
2", 6"	7.58 (1H, *m*)	7.99 (2H, *m*)
3",5"	-	7.38 (2H, *m*)
4"	8.08 (2H, *m*)	7.55 (1H, *m*)
	7.45 (2H, *m*)	
	7.58 (1H, *m*)	

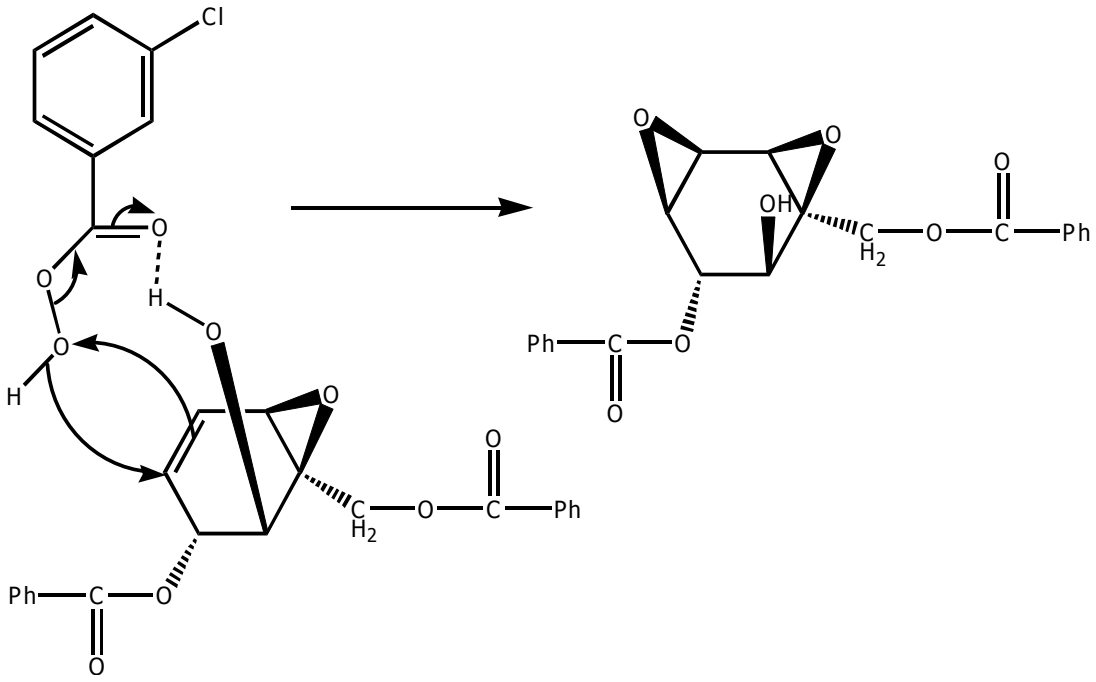
Table 57 **Comparison of ^{13}C NMR spectral data between** ST4 **and** diepoxide

Position	**Compound ST4, δ_C (ppm)**	**Diepoxide, δ_C (ppm)**
1	61.10	56.7
2	68.01	70.4
3	73.82	66.8
4	53.73	53.3
5	52.06	48.1
6	57.40	51.2
7	62.55	64.6
8	166.20	165.6
9	166.74	166.2
1'	129.30	129.2
2', 6' [a]	129.99	129.8
3',5' [b]	128.48	128.5
4' [c]	128.52	133.5
1"	129.30	129.0
2", 6" [a]	129.84	129.8
3",5" [b]	133.56	128.6
4" [c]	133.49	133.7

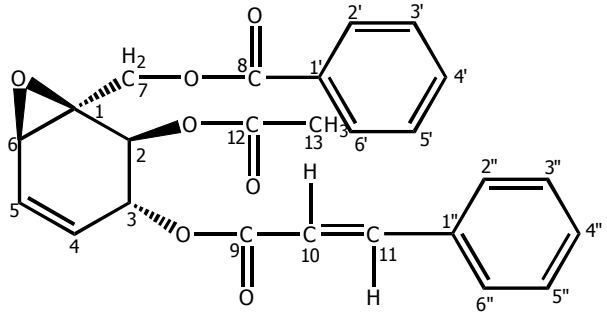
[a, b, c] May be interchangeable.

The epoxidation mechanism was influenced by an intermolecular H-bonding between the free OH of pipoxide (**SAH1**) and the carbonyl group of m-chloroperbenzoic acid which oriented the epoxidation to occure at the same phase to hydroxyl group, as shown in **scheme 14**.

Scheme 14 Epoxidation mechanism of compound **ST4**



2.5.5 **Compound ST5**



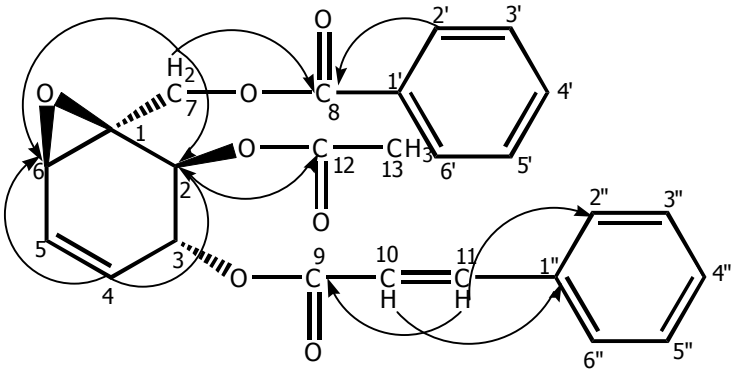
Acetylation reaction of compound **SAH2** with acetyl chloride in methylene chloride in the presence of trimethylamine at room temperature under nitrogen atmosphere for 6 days gave compound **SAH2** and **ST5** after PLC separation (silica gel, 25 % EtOAc : hexane). Compound **ST5** was a colorless viscous liquid, $[\alpha]_D^{26.3}$ = -83.3° (*c* = 0.012, $CHCl_3$). Their UV absorption spectrum (Fig. 90) showed maxima at 276, 235, 222 and 205 nm. The IR spectrum of compound **ST5** (Fig. 91) showed absorption bands at 1724 and 1719 cm^{-1} corresponding to the carbonyl groups.

The complete analysis of ^{13}C and ^{1}H NMR spectrum of compound **ST5** (see Table 61, Fig. 92 and 93) were assigned with the informations provided from ^{1}H-^{1}H COSY (see Table 59, Fig. 95), HMQC (Fig. 96) and HMBC (see Table 60, Fig.97). The ^{13}C NMR spectrum of compound **ST5** recorded in $CDCl_3$ (see Table 58, Fig. 93) showed 19 signals for 25 carbon atoms. Analysis of DEPT-90° and DEPT-135° (see Table 58, Fig. 94) spectra of this compound suggested the presence of one methyl carbon atom (δ 20.79), one methylene carbon atom (δ 62.23), thirteen signals of seventeen methine carbon atoms (δ 146.05, 133.63, 133.42, 130.60, 129.82, 128.91, 128.51, 128.25, 124.11, 117.06, 71.42, 71.18 and 54.50) and four signals of five quaternary carbon atoms (δ 170.24, 166.06 (2xC), 134.08 and 58.35)

Compound **ST5**, a derivative of compound **SAH2**, showed the same characteristic peaks in the IR and UV spectrum with those of compound **SAH2**. Comparison of the ^{1}H NMR spectrum (see Table 62) of the two compounds revealed their close structural similarity. Differences in the spectrum of compound **ST5** was shown as an acetoxy proton at δ 2.12 (3H, *s*) which was not observed in compound **SAH2**. The olefinic methine protons resonating at δ 7.70 (1H, *d*, *J* = 16 Hz), 6.41 (1H, *d*, *J* = 16 Hz), 6.09 (1H, *ddd*, *J* = 10, 4, 2.5 Hz) and 5.89 (1H, *dt*, *J* = 10, 2 Hz) could be assigned to H-11, H-10, H-5 and H-4, respectively. The coupling constant between H-11 (δ 7.70, 1H, *d*) and H-10 (δ 6.41, 1H, *d*) was 16 Hz, indicating that the two protons were *trans*-. The coupling constant between H-5 (δ 6.09, 1H, *ddd*, *J* = 10, 4, 2.5 Hz) and H-4 (δ 5.89, 1H, *dt*, *J* = 10, 2 Hz) was 10 Hz, indicating that the two protons were *cis*-. The doublet AB system of prochiral methylene proton appeared at δ 4.65 (1H, *d*, *J* = 12.5 Hz) and 4.41 (1H, *d*, *J* = 12 Hz). Ten methine aromatic protons resonated at δ 8.06 (2H, *dd*, *J* = 8.5, 1.5 Hz), 7.58 (1H, *m*), 7.52 (2H, *dd*, *J* = 6.5, 2.5 Hz), 7.47 (2H, *m*), 7.40 (2H, *m*) and 7.39 (1H, *m*) indicating that there were two monosubstituted phenyl rings in this compound. The epoxy methine proton and two oxymethine protons appeared at δ 3.61 (1H, *dd*, *J* = 4, 2 Hz), 5.78 (1H, *d*, *J* = 8.5 Hz) and 5.70 (1H, *ddd*, *J* = 8.5, 2.5, 1.5 Hz) which were assigned to H-6, H-2 and H-3, respectively. The H-2 (δ 5.78, 1H, *d*, *J* = 8.5 Hz) of compound **ST5** appeared at the lower field than H-2 (δ 4.25, 1H, *d*, *J* = 8.1 Hz) of compound **SAH2**. These observations indicated that the 2-position should be connected with the acetyl group.

The HMBC correlation of compound **ST5** were similar to compound **SAH2** except H-2 of compound **ST5** (δ 5.78, 1H, *d*, *J* = 8.5 Hz) showed correlation peaks with C-2-C=O (δ 170.24) and C-3 (δ 71.42), thus confirmed the position of the acetoxy group at C-2 (δ 71.18). From NOE experiment (Fig. 98 and 99), irradiation of H-7b (δ 4.65) showed enhancement of H-7a (δ 4.41). Irradiation of H-7a (δ 4.41) showed the enhancement of H-7b (δ 4.65) and H-6 (δ 3.61). Irradiation of H-

6 (δ 3.61) showed the enhancement of H-7a (δ 4.4 1) and H-5 (δ 6.09). Irradiation of H-3 (δ 5.70) showed the enhancement of H-4 (δ 5.89). Irradiation of H-2 (δ 5.78) showed the enhancement of H-7b (δ 4.6 5) and no enhancement was observed on H-3 (δ 5.70). Thus, this compound should be benzoyloxymethyl and cinnamoyl groups on the same side at position 1 and 3, respectively and opposite side to acetoxyl group at position 2. These observations indicated that both compounds are derivatives. Thus, compound **ST5** was identified as cinnamoxide acetate.



Selected HMBC Correlation

Table 58 ^{13}C and DEPT spectral data of compound **ST5**

δ_C	**DEPT-90°**	**DEPT-135°**	**Type of carbon**
170.24			C
166.06			C
146.05	146.05	146.05	CH
134.08			C
133.63	133.63	133.63	CH
133.42	133.41	133.41	CH
130.60	130.60	130.95	CH
129.82	129.81	129.81	CH
128.91	128.91	128.91	CH
128.51	128.50	128.52	CH
128.25	128.25	128.25	CH
124.11	124.11	124.11	CH
117.06	117.05	117.05	CH
71.42	71.42	71.42	CH
71.18	71.17	71.18	CH
62.23		62.23	CH_2
58.35			C
54.50	54.49	54.49	CH
20.79		20.78	CH_3

Table 59 500 MHz COSY correlation of some protons of compound **ST5**

δ_H (ppm)	Proton Correlation with δ_H (ppm)
H-2 (5.78)	H-3 (5.70)
H-3 (5.70)	H-2 (5.78), H-4 (5.89), H-5 (6.09)
H-4 (5.89)	H-3 (5.70), H-5 (6.09), H-6 (3.61)
H-5 (6.09)	H-3 (5.70), H-4 (5.89), H-6 (3.61)
H-6 (3.61)	H-4 (5.89), H-5 (6.09)
H-7a (4.41)	H-7b (4.65)
H-10 (6.41)	H-11 (7.70)
H-2', H-6', (8.06)	H-3', H-5' (7.40), H-4' (7.58)
H-2", H-6"(7.52)	H-3", H5" (7.40)
H-3', H-5' (7.47)	H-2', H-6' (8.06), H-4' (7.58)
H-3", H5" (7.40)	H-2", H-6" (7.52), H-4" (7.39)
H-4' (7.58)	H-2', H-6' (8.06), H-3', H-5' (7.47)
H-4" (7.39)	H-3", H5" (7.40)

Table 60 Major HMBC correlation of compound **ST5**

Position	δ_H (ppm)	δ_C (ppm)
1	-	-
2	5.78 (1H, *d*, *J* = 8.5 Hz)	C-3 (71.42) and C-12 (170.24)
3	5.70 (1H, *ddd*, *J* = 8.5, 2.5, 1.5 Hz)	C-2 (71.18) and C-4 (133.63)
4	5.89 (1H, *dt*, *J* = 10, 2 Hz)	C-2 (71.18) and C-6 (54.50)
5	6.09 (1H, *ddd*, *J* = 10, 4, 2.5 Hz)	-
6	3.61 (1H, *dd*, *J* = 4, 2 Hz)	C-5 (124.11)

Table 60 (Continued)

Position	δ_H (ppm)	δ_C (ppm)
7	4.41 (1H, *d* (AB), *J* = 12 Hz) 4.65 (1H, *d* (AB), *J* = 12 Hz)	C-1 (58.35), C-2 (71.18), C-3 (71.42), C-6 (54.50) and C-8 (166.06)
8	-	-
9	-	-
10	6.41 (1H, *d*, *J* = 16 Hz)	C-1" (134.08) and C-9 (166.06)
11	7.70 (1H, *d*, *J* = 16 Hz)	C-2", C-6" (128.25), C-9 (166.06) and C-10 (117.06)
12	-	-
13	2.12 (3H, *s*)	C-12 (170.24)
1' / 1"	-	-
2', 6'	8.06 (2H, *dd*, *J* = 8.5, 1.5 Hz)	C-8 (166.06), C-1' (134.08) and C-4' (133.42)
3', 5'	7.47 (2H, *m*)	-
4'	7.58 (1H, *m*)	C-2', C-6' (129.82)
2", 6"	7.52 (2H, *dd*, *J* = 6.5, 2.5 Hz)	C-4" (130.60)
3", 5"	7.40 (2H, *m*)	C-1" (134.08)
4"	7.39 (1H, *m*)	C-3", C-5" (128.91)

Table 61 ^{1}H and ^{13}C NMR spectral data of compound **ST5**

Position	δ_C [#] (ppm)		δ_H (ppm)
1	58.35	C	-
2	71.18	CH	5.78 (1H, *d*, *J* = 8.5 Hz)
3	71.42	CH	5.70 (1H, *ddd*, *J* = 8.5, 2.5, 1.5 Hz)
4	133.63	CH	5.89 (1H, *dt*, *J* = 10, 2 Hz)
5	124.11	CH	6.09 (1H, *ddd*, *J* = 10, 4, 2.5 Hz)
6	54.50	CH	3.61 (1H, *dd*, *J* = 4, 2 Hz)
7	62.23	CH_2	4.41 (1H, *d* (AB), *J* = 12 Hz)
			4.65 (1H, *d* (AB), *J* = 12 Hz)
8	166.06	C	-
9	166.06	C	-
10	117.06	CH	6.41 (1H, *d*, *J* = 16 Hz)
11	146.05	CH	7.70 (1H, *d*, *J* = 16 Hz)
12	170.24	C	-
13	20.79	CH_3	2.12 (3H, *s*)
1'	134.08	C	-
2', 6'	129.82	CH	8.06 (2H, *dd*, *J* = 8.5, 1.5 Hz)
3', 5'	128.51	CH	7.47 (2H, *m*)
4'	133.42	CH	7.58 (1H, *m*)
1"	134.08	C	-
2", 6"	128.25	CH	7.52 (2H, *dd*, *J* = 6.5, 2.5 Hz)
3", 5"	128.91	CH	7.40 (2H, *m*)
4"	130.60	CH	7.39 (1H, *m*)

[#] Carbon type deduced from DEPT experiment.

Table 62 Comparison of ^{1}H NMR spectral data between compound **ST5** and **SAH2**

Position	Compound ST5, δ_H (ppm)	Compound SAH2, δ_H (ppm)
1	-	-
2	5.78 (1H, *d*, *J* = 8.5 Hz)	4.25 (1H, *d*, *J* = 8.1 Hz)
3	5.70 (1H, *ddd*, *J* = 8.5, 2.5, 1.5 Hz)	5.56 (1H, *dt*, *J* = 8.1, 2.1 Hz)
4	5.89 (1H, *dt*, *J* = 10, 2 Hz)	5.86 (1H, *dt*, *J* = 9.9, 1.8 Hz)
5	6.09 (1H, *ddd*, *J* = 10, 4, 2.5 Hz)	6.08 (1H, *ddd*, *J* = 9.9, 3.6, 2.7 Hz)
6	3.61 (1H, *dd*, *J* = 4, 2 Hz)	3.59 (1H, *dd*, *J* = 3.9, 1.8 Hz)
7	4.41 (1H, *d* (AB), *J* = 12 Hz)	4.48 (1H, *d* (AB), *J* = 12 Hz)
	4.65 (1H, *d* (AB), *J* = 12 Hz)	5.00 (1H, *d* (AB), *J* = 12 Hz)
8	-	-
9	-	-
10	6.41 (1H, *d*, *J* = 16 Hz)	6.47 (1H, *d*, *J* = 15.9 Hz)
11	7.70 (1H, *d*, *J* = 16 Hz)	7.76 (1H, *d*, *J* = 15.9 Hz)
12	-	-
13	2.12 (3H, *s*)	-
1'	-	-
2', 6'	8.06 (2H, *dd*, *J* = 8.5, 1.5 Hz)	8.06 (2H, *m*)
3', 5'	7.47 (2H, *m*)	7.46 (2H, *m*)
4'	7.58 (1H, *m*)	7.59 (1H, *m*)
1"	-	-
2", 6"	7.52 (2H, *dd*, *J* = 6.5, 2.5 Hz)	7.53 (2H, *m*)
3", 5"	7.40 (2H, *m*)	7.40 (2H, *m*)
4"	7.39 (1H, *m*)	7.39 (1H, *m*

Table 63 Comparison of ^{13}C NMR spectral data between compound **ST5** and **SAH2**

Position	Compound ST5, δ_C (ppm)	Compound SAH2, δ_C (ppm)
1	58.35	59.55
2	71.18	71.10
3	71.42	74.40
4	133.63	133.12
5	124.11	124.64
6	54.50	54.25
7	62.23	62.92
8	166.06	166.20
9	166.06	167.32
10	117.06	117.21
11	146.05	146.31
12	170.24	-
13	20.79	-
1'	134.08	129.51
2', 6'	129.82	129.83
3', 5'	128.51	128.51
4'	133.42	133.43
1"	134.08	134.11
2", 6"	128.25	128.25
3", 5"	128.91	128.97
4"	130.60	130.66

3.3 Biological activities of the crude extract, pure compound from U. purpurea Blume and synthetic pipoxide derivative

The biological activity of crude hexane extract exhibited moderate activity against NCI-H187 cell line. It also showed activity against *Mycobacterium tuberculosis* H-37Ra and vero cells but no activity against *Staphylococcus aureus* ATCC15923, *S. aureus* SK1 and fungi. The crude methylene chloride extract showed moderate activity against fungal and weak activity against vero cells but no activity against NCI-H187 cell line. The crude methanol extract showed only activity against vero cells. The pure compounds (**SAH1, SAH3, ST1** and **ST2)** exhibited no activity against *Staphylococcus aureus* ATCC15923 and *S. aureus* SK1, whereas vancomycin, a standard antibiotic, showed activity against *Staphylococcus aureus* ATCC15923 and *S. aureus* SK1 with MIC value 2 µg/ml and IZ value of 16.65 mm. The results were summarized in **Table 64**.