INTRODUCTION

Diarylheptanoids are an important class of structurally distinctive natural plant metabolites containing the 1,7-diphenylheptane moiety. More than 400 diarylheptanoids have been isolated from natural sources since first diarylheptanoid identified in 1815.\(^1\) This kind of diarylheptanoids is mainly isolated in the *Zingiber, Curcuma, Alpinia, Viscum, Alnus* and *Myrica* etc.\(^2\) They are known to have recognized as potential therapeutic agents for its numerous physiological activity such as anti-inflammatory, antioxidant, antitumor, estrogenic, leishmanicidal, melanogenesis, hepatoprotective and neuroprotective activities.

**Yashabushidiol**

Hashimoto *et al.* isolated five new diarylheptanoids (1-5) from the male flowers of *Alnus sieboldiana* Matsum.\(^3\) Yashabushiketols 6 and 7 was first extracted in 1970 from young shoots of the plant *Alnus sieboldiana* and its structure and absolute configuration were determined later.\(^4\)

![Yashabushidiol A (1)](image1)

![Yashabushidiol B (2)](image2)

![Yashabushiketodiol A (3)](image3)

![Yashabushiketodiol B (4)](image4)

![Yashabushitriol (5)](image5)

![\((R)\) Yashabushiketol (6)](image6)

![\((S)\) Yashabushiketol (7)](image7)

Ali *et al.* isolated three diarylheptanoids (8-10) from an EtOH extract of the seeds of *Alpinia blepharocalyx*.\(^5\) In bioassays these diarylheptanoids exhibit significant
antiproliferative activity against murine colon 26-L5 carcinoma and human HT-1080 fibrosarcoma cells. Later in 2013 structurally related diarylheptanoids (11-13) isolated from the leaves and twigs of *Viscum album* by Nhiem *et al.* and these compounds shows significant anti-inflammatory activity on LPS-stimulated production of TNF-α, IL-6 and IL-12p40 with IC$_{50}$ values ranging from 0.09 ± 0.01 to 11.84 ± 0.34 μM.$^6$

Kikuzaki *et al.* isolated two diarylheptanoids and acetylated diarylheptanoids (14-19) from the rhizomes of *Zingiber officinale*.$^7$

Yokosuka *et al.* isolated two diarylheptanoids (20, 21) and six diarylheptanoid glucosides (22-27) from the rhizomes of *Tacca chantrieri*.$^8$ These diarylheptanoids were
tested for cytotoxic activity against HL-60 human promyelocytic leukemia cells, HSC-2 human oral squamous carcinoma cells and normal human gingival fibroblasts (HGF). The IC$_{50}$ values are in the range of 1.8-54 μg/mL were found.

**PRESENT WORK**

Diarylheptanoids are a family of natural plant metabolites whose characteristic feature is the presence of two aromatic rings tethered by a linear seven-carbon chain. The linear diarylheptanoids having 1,3-diol system exhibit various biological activities such as antioxidative, hepatoprotective, antiproliferative, anti-inflammatory and antiemetic activities. Rhoiptelol C (28), a linear diarylheptanoid, was isolated from the fruits of *Rhoiptelea chiliantha* belongs to the Zingiberaceae family. It was also isolated from the stems of *Engelhardia roxburghiana* in 2012 by Wu and coworkers. The structure elucidation was mainly based on spectroscopic analysis and the stereochemistry was proved through Moshers ester analysis method. Due to the interesting structure with three chiral hydroxyl groups and our continuing interest in total synthesis of naturally
isolated linear diarylheptanoids, herein we wish to report the first synthesis of diarylheptanoid Rhoiptelol C 28.

**Retro synthetic strategy:**

The retrosynthetic analysis is reported in Scheme 1. We envisaged that the target diarylheptanoid, Rhoiptelol C (28) could be prepared through a sharples asymmetric dihydroxylation of compound 36. The compound 36 could be prepared from the cross metathesis of chiral alcohol 33 and chavicol 34. Chiral alcohol 33 could be made by Wittig homologation and Keck’s asymmetric allylation reaction from the commercially available vanilline 29.

![Scheme 1](image)

The vanilline 29 was subjected to 2Carbon-Wittig homologation with (carboethoxymethylene)triphenylphosphorane in benzene under reflux conditions to afford the α,β-unsaturated ester 30 in 96% yield. The structure 30 was confirmed by its $^1$H NMR spectrum (Fig. 1.2.01) in which double bond protons appeared at δ 7.62 (d, $J = 15.8$ Hz, 1H) and 6.29 (d, $J = 15.8$ Hz, 1H) respectively. IR spectrum showed absorption band at 1702 cm$^{-1}$ for ester carbonyl group. Its mass spectrum (Fig. 1.2.03) showed a molecular ion peak at m/z 245 [M+Na]$^+$ further confirmed the product 30 (Scheme 2).

![Scheme 2](image)
The α,β-unsaturated ester 30 was reduced by using NiCl₂·6H₂O, NaBH₄ in MeOH to give the saturated ester 31 in 93% yield. The formation of compound 31 was confirmed by its ¹H NMR spectrum (Fig. 1.2.04) which showed a conspicuous disappearance of double bond protons and appearance of two triplets at δ 2.85 (t, J = 7.5 Hz, 2H), δ 2.54 (t, J = 7.5 Hz, 2H) for correspondig two -CH₂ groups of compound 31. IR spectrum showed absorption band at 1739 cm⁻¹ due to saturated ester group. The structure of the compound 31 was further confirmed by its mass spectrum (Fig. 1.2.06), which showed molecular ion peak at m/z 247 [M+Na]⁺ (Scheme 3).

Scheme 3

The hydroxy group in compound 31 protected with P-TsCl and triethylamine in CH₂Cl₂ gave tosylester 32 in 91% yield. The ¹H NMR spectrum (Fig. 1.2.07), which displays signals at δ 7.73 (d, J = 8.3 Hz, 2H), 7.27 (d, J = 8.3 Hz, 2H) and 2.45 (s, 3H) for the tosyl protons and the mass spectrum (Fig. 1.2.10) showed a peak at m/z 396 [M+NH₄]⁺ further confirmed the product 32 (Scheme 4).

Scheme 4

The compound 32 was then reduced to the corresponding aldehyde using DIBAL-H in dry CH₂Cl₂ at -78 °C in 92% yield. The unstable aldehyde was used immediately for the next reaction without further purification (Scheme 5).

The aldehyde was subjected to Keck’s asymmetric allylation with allyl-n-tributyl stannane in the presence of (R)-Binol and Ti(OiPr)₄ in dry CH₂Cl₂ at -78 °C to -20 °C to furnish chiral allylic alcohol 33 in 89% yield. The compound 33 was characterized from its ¹H NMR spectrum (Fig. 1.2.11) which showed multiplet at δ 5.88-5.72 (m, 1H) and 5.15 (dd, J = 10.9, 5.3 Hz, 2H) for olefinic group and a multiplet
at $\delta$ 3.69-3.58 (m, 1H) for newly created proton attached to -OH. IR spectrum (Fig. 1.2.13) showed absorption band at 3422 cm$^{-1}$ due to hydroxyl functional group and the molecular ion peak at $m/z$ 399 [M+Na]$^+$ in its mass spectrum (Fig. 1.2.14), confirmed the formation of allylation product 33 (Scheme 5).

**Scheme 5**

Next the attention was directed towards the olefin cross metathesis of compound 33 with chavicol 34 by using Grubbs 2nd generation catalyst in dry CH$_2$Cl$_2$ to afford 35 in 81% yield.$^{13}$ The formation of compound 35 was confirmed by its $^1$H NMR spectrum (Fig. 1.2.15) which showed two doublet of triplets at $\delta$ 5.64 (dt, $J$ = 15.1, 6.6 Hz, 1 H), 5.42 (dt, $J$ = 15.1, 7.1 Hz, 1H) for the double bond protons. IR spectrum (Fig. 1.2.17) shows absorption band at 3452 cm$^{-1}$ for hydroxyl functional group and its mass spectrum (Fig. 1.2.18) showed a molecular ion peak at $m/z$ 505 [M+Na]$^+$ further confirmed the structure of compound 35 (Scheme 6).

**Scheme 6**

Now, the tosyl group in compound 35 was removed employing K$_2$CO$_3$ in MeOH at reflux temperature for 2 h to give compound 36 in 80% yield. The formation of compound 36 was identified by its $^1$H NMR (Fig. 1.2.19), $^{13}$C NMR (Fig. 1.2.20) spectrum in which disappearance of peaks corresponding to tosyl group was observed.
and all other protons resonated at the expected chemical shift values. IR spectrum (Fig. 1.2.21) showed a strong absorption band at 3413 cm\(^{-1}\) due to hydroxyl group. Its mass spectrum (Fig. 1.2.22) shows a molecular ion peak at \(m/z\) 351 \([M+Na]^+\) which further confirmed the product 36 (Scheme 7).

\[
\text{Scheme 7}
\]

Finally, the compound 45 subjected to sharples asymmetric dihydroxylation\(^{14}\) using AD-mix-\(\alpha\) and methane sulfonamide in \(t\)-BuOH-H\(_2\)O (1:1) at 0 \(^\circ\)C to afford the target molecule Rhoiptelol C 28 in 75\% yield (Scheme 8). The formation of product was established by the study of their \(^1\)H NMR (Fig. 1.2.23), \(^{13}\)C NMR (Fig. 1.2.24), IR (Fig. 1.2.25) and ESI-MS spectral data and found to be identical in all respects that reported for the natural product.

\[
\text{Scheme 8}
\]

Conclusion

In conclusion, we have described an efficient and economic route for the total synthesis of Rhoiptelol C (28). The synthesis involved Keck’s asymmetric allylation reaction, olefin cross metathesis and Sharples asymmetric dihydroxylation as key steps.

**EXPERIMENTAL**

**EXPERIMENTAL DATA:**

\((E)\)-ethyl 3-(4-hydroxy-3-methoxyphenyl)acrylate (30):
To a solution of the aldehyde 29 (4 g, 26.28 mmol) in dry benzene (40 mL) was added \( \text{Ph}_3\text{P} = \text{CHCO}_2\text{Et} \) (10.97 g, 31.54 mmol) and stirred at reflux temperature for 1 h. After completion of the reaction, the solvent was removed under reduced pressure and the residue purified by column chromatography (EtOAc:Hexanes, 95:5) to afford 30 as colorless syrup (5.60 g, 96%) as a mixture of geometrical isomers (E/Z).

\(^1\text{H NMR (300 MHz, CDCl}_3\) : \( \delta \) 7.62 (d, \( J = 15.8 \) Hz, 1H), 7.12-7.01 (m, 2H), 6.92 (d, \( J = 8.1 \) Hz, 1H), 6.29 (d, \( J = 15.8 \) Hz, 1H), 4.26 (q, \( J = 7.1 \) Hz, 2H), 3.92 (s, 3H), 1.34 (t, \( J = 7.1 \) Hz, 3H). (Fig. 1.1.03).

\(^{13}\text{C NMR (75 MHz, CDCl}_3\) : \( \delta \) 167.2, 147.9, 146.7, 144.6, 126.9, 122.9, 115.4, 114.7, 109.3, 60.2, 55.8, 14.2.

\( \text{IR (neat) cm}^{-1} \) : 3421, 1712, 1638, 1223, 1132.

\( \text{ESIMS (m/z)} \) : 245 [M + Na]^+.

**Molecular formula** : \( \text{C}_{12}\text{H}_{14}\text{O}_4\).

**Ethyl 3-(4-hydroxy-3-methoxyphenyl)propanoate (31):**

To a stirred solution of conjugated alkene 30 (5 g, 22.52 mmol) in MeOH (100 mL), \( \text{NiCl}_4\cdot6\text{H}_2\text{O} \) (1.06 g, 4.50 mmol) was added at 0 °C. The resulting mixture was stirred at 0 °C for 10 min and \( \text{NaBH}_4 \) (1.71 g, 45.04 mmol) was added in small portions. The reaction mixture was stirred for another 1 h and quenched with water. The mixture was filtered through Celite pad, washed with EtOAc. The whole filtrate concentrated under vacuo, diluted with \( \text{H}_2\text{O} \) and extracted with EtOAc. The organic extract was washed with brine and dried over anh. \( \text{Na}_2\text{SO}_4 \) and evaporated. The crude reaction mixture was purified by silicagel column chromatography (EtOAc:Hexanes, 25:75) to
Chapter I Section B  First Stereoselective Total synthesis of Rhoiptelol C

provide the corresponding saturated ester compound 31 (4.69 g, 93%) as a colourless liquid.

$^1$H NMR (300 MHz, CDCl$_3$) : $\delta$ 6.77 (d, $J = 8.3$ Hz, 1H), 6.67-6.62 (m, 2H), 4.1 (q, $J = 7.5$ Hz, 2H), 3.88 (s, 3H), 2.85 (t, $J = 7.5$ Hz, 2H), 2.54 (t, $J = 7.5$ Hz, 2H), 1.24 (t, $J = 7.5$ Hz, 3H).

$^{13}$C NMR (75 MHz, CDCl$_3$) : $\delta$ 172.9, 146.3, 143.8, 132.3, 120.6, 114.2, 110.8, 60.3, 55.6, 36.2, 30.5, 14.0.

IR (neat) cm$^{-1}$ : 3448, 2951, 1732, 1517, 1271, 1236, 1205, 1155, 1034, 819.

ESIMS (m/z) : 247 [M+Na]$^+$.  

Molecular formula : C$_{12}$H$_{16}$O$_4$.

Ethyl 3-(3-methoxy-4-(tosyloxy)phenyl)propanoate (32):

To a stirred solution of hydroxy ester 31 (4.5 g, 20.08 mmol) in CH$_2$Cl$_2$ (50 mL) was added NEt$_3$ (2.2 g, 20.08 mmol) at 0 $^\circ$C, followed by the addition of $p$-toluenesulfonyl chloride (3.83 g, 20.08 mmol). Then, the reaction was stirred at room temperature for 2 h. The reaction was diluted with 1 N HCl (20 mL) and the layers were separated. The organic layer was washed with saturated aqueous NaHCO$_3$ (20 mL) and brine solution (20 mL). The combined organic layers were dried with anh. Na$_2$SO$_4$, and concentrated under reduced pressure. The crude residue was purified by silicagel column chromatography (EtOAc:Hexanes, 1:9) to give 32 (6.91 g, 91%) as a colorless viscous liquid.

$^1$H NMR (300 MHz, CDCl$_3$) : $\delta$ 7.73 (d, $J = 8.3$ Hz, 2 H), 7.27 (d, $J = 8.3$ Hz, 2H), 7.0 (d, $J = 8.1$ Hz, 1H), 6.72-6.63 (m, 2H), 4.1 (q, $J = 7.1$ Hz, 2H), 3.56 (s, 3H), 2.88 (t, $J = 7.5$ Hz, 2H), 2.56 (t, $J = 7.5$ Hz, 2H), 2.45 (s, 3H), 1.23 (t, $J = 7.1$ Hz, 3H).
Chapter I Section B  First Stereoselective Total synthesis of Rhoiptelol C

$^{13}$C NMR (75 MHz, CDCl$_3$): $\delta$ 172.5, 151.5, 144.8, 140.8, 136.7, 133.2, 129.2, 128.4, 123.7, 120.1, 112.7, 60.4, 55.4, 35.6, 30.7, 21.5, 14.1.

IR (neat) cm$^{-1}$: $\delta$ 2980, 1731, 1599, 1506, 1371, 1178, 1152, 849, 817.

ESIMS ($m/z$): 396 [M + NH$_4$]$^+$.

Molecular formula: C$_{19}$H$_{22}$O$_6$S.

(S)-4-(3-hydroxyhex-5-en-1-yl)-2-methoxyphenyl 4-methylbenzenesulfonate (33):

To a cooled (-78 °C) stirred solution of ester compound 32 (2.5 g, 6.61 mmol) in dry CH$_2$Cl$_2$ (50 mL) was added DIBAL-H (1.0 M, 6.61 mL, 6.61 mmol) and the reaction was stirred for 0.5 h at -78 °C. After completion of the reaction, the reaction was quenched with saturated sodium potassium tartarate (25 mL) and stirred for 0.5 h. The reaction mixture was then extracted into CH$_2$Cl$_2$ (3 x 75 mL). The combined organic phase was washed with brine, and dried over anh. Na$_2$SO$_4$, and concentrated in vacuo, to give crude aldehyde (2.03 g, 92%). The crude product was directly used for the next step.

To the stirred solution of oven-dried 4-Å molecular sieves (3 g) in CH$_2$Cl$_2$ (30 mL) under N$_2$ atmosphere, was added (R)-BINOL (0.214 g, 0.75 mmol) and Ti(OiPr)$_4$ (0.23 mL, 0.75 mmol). The reaction mixture was heated at reflux temperature for a period of 3 h, and then allowed to cool to room temperature. The crude aldehyde (1.25g, 3.74 mmol) in CH$_2$Cl$_2$ (10 mL), was added to the reaction mixture. After the mixture stirred for 0.5 h at room temperature, cooled to -78 °C, allyltributyltin (1.5 mL, 4.86 mmol) was added slowly. The reaction mixture was stirred for an additional 10 min at -78 °C, then the reaction was stirred at -20 °C for 24 h, the reaction mixture was quenched with saturated aqueous NaHCO$_3$ solution (50 mL) and filtered through Celite pad, then the two layers were separated. The aqueous layer was extracted with CH$_2$Cl$_2$ (2 x 20 mL). The combined organic layers were washed with brine (20 mL), dried with
anh. Na₂SO₄, and concentrated under reduced pressure to give the crude product. The crude residue was purified by column chromatography (EtOAc:hexane, 2:8) to give 33 (1.25 g, 89%; 96% ee) as a viscous liquid: The ee was determined by chiral HPLC column: (CHIRAL PAK IA: 250 x 4.6mm, 5μ) mobile phase: 20% IPA in Hexane, Flow rate: 1.0 mL/min, detection: 210 nm, Ret. Time: 12.406 min, 96% ee.

\[ [\alpha]_D^{25} = -4.0 \ (c \ 0.2, \text{CHCl}_3). \]

**¹H NMR (300 MHz, CDCl₃)**: δ 7.75 (d, J = 8.3 Hz, 2H), 7.29 (d, J = 7.9 Hz, 2H), 7.01 (d, J = 8.1 Hz, 1H), 6.73-6.64 (m, 2H), 5.88-5.72 (m, 1H), 5.15 (dd, J = 10.9, 5.3 Hz, 2H), 3.69-3.58 (m, 1H), 3.56 (s, 3H), 2.83-2.70 (m, 1H), 2.69-2.56 (m, 1H), 2.45 (s, 3H), 2.37-2.25 (m, 1H), 2.22-2.09 (m, 1H), 1.79-1.68 (m, 2H).

**¹³C NMR (75 MHz, CDCl₃)**: δ 151.4, 144.8, 142.4, 136.4, 134.4, 133.3, 129.2, 128.5, 123.6, 120.2, 118.4, 112.8, 69.7, 55.4, 42.0, 38.1, 31.9, 21.6.

**IR (neat) cm⁻¹**: 3422, 2926, 1599, 1505, 1369, 1176, 1090, 851.

**ESIMS (m/z)**: 399 [M + Na]⁺.

**Molecular formula**: C₂₀H₂₄O₅S.

(S,E)-4-(3-hydroxy-7-(4-hydroxyphenyl)hept-5-en-1-yl)-2-methoxyphenyl 4-methylbenzenesulfonate (35):

![Chemical Structure](35.png)

To a solution of 33 (150 mg, 0.39 mmol) in CH₂Cl₂ (150 mL) was added Grubbs’ second generation catalyst (17 mg, 0.02 mmol) and chavicol 34 (267 mg, 1.99 mmol) at room temperature and the mixture was stirred at 40 °C for 2 h. The reaction mixture was concentrated in vacuo to give the crude product, which was purified by column chromatography (EtOAc:Hexanes, 4:6) to give desired compound 35 (155 mg, 81%) as a colorless viscous liquid.

\[ [\alpha]_D^{25} = -9.0 \ (c \ 0.2, \text{CHCl}_3). \]
First Stereoselective Total synthesis of Rhoiptelol C

1H NMR (300 MHz, CDCl3) : δ 7.74 (d, J = 8.3 Hz, 2H), 7.27 (d, J = 9.4 Hz, 2H), 7.0-6.88 (m, 3H), 6.68-6.58 (m, 4H), 5.63 (dt, J = 15.1, 6.6 Hz, 1H), 5.42 (dt, J = 15.1, 7.1 Hz, 1H), 3.68-3.57 (m, 1H), 3.52 (s, 3H), 3.24 (d, J = 6.6 Hz, 2H), 2.80-2.66 (m, 1H), 2.64-2.51 (m, 1H), 2.44 (s, 3H), 2.35-2.09 (m, 2H), 1.79-1.63 (m, 2H).

13C NMR (75 MHz, CDCl3) : δ 154.1, 151.4, 144.9, 142.4, 136.3, 133.8, 133.1, 132.0, 129.4, 129.3, 128.5, 126.4, 123.6, 120.2, 115.2, 112.8, 70.3, 55.4, 40.6, 38.1, 38.0, 31.8, 21.6

IR (neat) cm⁻¹ : 3452, 2926, 1599, 1509, 1366, 1175, 1090, 851, 817.

ESIMS (m/z) : 505 [M + Na]^+.

Molecular formula : C27H30O6S.

(S,E)-4-(3-hydroxy-7-(4-hydroxyphenyl)hept-5-en-1-yl)-2-methoxyphenol (36):

To a solution of compound 35 (90 mg, 0.18 mmol) in MeOH (20 mL) was added K2CO3 (150 mg, 1.08 mmol) and the mixture was heated at reflux temperature for 2 h, cooled to 0 °C, acidified with 1 M HCl until pH 2. The combined solution was extracted with EtOAc (3 x 20 mL), washed with brine (15 mL), dried over anh. Na2SO4, and concentrated under vacuo. Purification by column chromatography (EtOAc:hexane, 6:4) afforded 36 (49 mg, 80%) as colorless viscous liquid.

[α]D²⁵ : -16.50 (c 0.2, CHCl3).

1H NMR (300 MHz, CDCl3) : δ 7.02 (d, J = 8.3 Hz, 2H), 6.83 (d, J = 7.5 Hz, 1H), 6.78-6.64 (m, 4H), 5.69 (dt, J = 15.1, 6.7 Hz, 1H), 5.48 (dt, J = 15.1, 7.5 Hz, 1H), 3.86 (s, 3H), 3.71-3.60 (m, 1H), 3.29 (d, J = 6.8 Hz, 2H), 2.79-2.54 (m, 2H), 2.28-2.23 (m, 1H), 2.21-2.09 (m, 1H), 1.81-1.70 (m, 1H).
**Chapter I Section B**  

**First Stereoselective Total synthesis of Rhoiptelol C**

| **13C NMR (75 MHz, CDCl₃)** | δ 154.0, 146.3, 143.5, 133.9, 133.6, 132.1, 129.4, 126.6, 120.8, 115.2, 114.2, 111.0, 70.3, 55.8, 40.5, 38.5, 38.1, 31.5. |
| **IR (neat) cm⁻¹** | 3413, 2926, 1513, 1266, 1232, 1152, 1032, 770. |
| **ESIMS (m/z)** | 351 [M+ Na]⁺. |
| **Molecular formula** | C₂₀H₂₄O₄. |
| **(2S,3S,5S)-7-(4-hydroxy-3-methoxyphenyl)-1-(4-hydroxyphenyl)heptane-2,3,5-triol (28):** | ![Structure of 28] |

AD-mix-α (42.6 mg, 1.4 g/mm) and methanesulfonamide (39 mg, 4.2 mmol) were added to t-BuOH:H₂O (10 mL, 1:1), the mixture was stirred at r.t. for 10 min and then cooled to 0 °C. To this soln was added alkene 36 (10 mg, 0.03 mmol) and the mixture stirred for 24 h at 0 °C. After completion of the reaction, the soln was quenched with solid Na₂SO₃ (10 g) at r.t. and diluted with EtOAc (50 mL). The aqueous layer was extracted with EtOAc (3 x 10 mL) and the combined organic layer washed with brine (10 mL) and dried over anh. Na₂SO₄. After evaporation of the solvent, the residue was purified by silicagel column chromatography (EtOAc:hexane, 3:7) to afford 28 (8 mg, 75%) as white solid.

[α]D²⁵ : -21 (c 0.4, MeOH).

| **1H NMR (300 MHz, Me₂CO-d₆)** | δ 7.07 (d, J = 8.1 Hz, 2H), 6.80 (d, J = 1.9 Hz, 1H), 6.73 (d, J = 8.1 Hz, 2H), 6.71 (d, J = 8.1 Hz, 2H), 6.63 (dd, J = 8.1, 1.9 Hz, 1H), 3.81 (m, 1H), 3.80 (s, 3H), 3.73-3.52 (m, 2H), 2.79 (dd, J = 13.7, 4.9 Hz, 1H), 2.71-2.50 (m, 2H), 1.76-1.60 (m, 4H). |
| **13C NMR (75 MHz, Me₂CO-d₆)** | δ 156.4, 148.0, 145.2, 134.6, 131.1, 121.3, 115.7, 115.5, 112.7, 76.1, 73.7, 71.0, 56.0, 40.9, 40.4, 39.3, 31.9. |
| **IR (KBr) cm⁻¹** | 3413, 2926, 1513, 1266, 1232, 1152, 1032, 770. |
Chapter I Section B  First Stereoselective Total synthesis of Rhoiptelol C

ESIMS (m/z) : 385 [M+ Na]+.
Molecular formula : C_{20}H_{26}O_{6}.

REFERENCES:

Chapter I Section B
First Stereoselective Total synthesis of Rhoiptelol C

Fig. 1.2.01: $^1$H NMR (300 MHz, CDCl$_3$) spectrum of compound 30

Fig. 1.2.02: $^{13}$C NMR (75 MHz, CDCl$_3$) spectrum of compound 30
Chapter I Section B  First Stereoselective Total synthesis of Rhoiptelol C

Fig. 1.2.03: ESI-MS spectrum of compound 30

Fig. 1.2.04: $^1$H NMR (300 MHz, CDCl$_3$) spectrum of compound 31
First Stereoselective Total synthesis of Rhoiptelol C

Fig. 1.2.05: $^{13}$C NMR (75 MHz, CDCl$_3$) spectrum of compound 31

Fig. 1.2.06: ESI-MS spectrum of compound 31
Chapter I Section B  
First Stereoselective Total synthesis of Rhoiptelol C

Fig. 1.2.07: $^1$H NMR (300 MHz, CDCl$_3$) spectrum of compound 32

Fig. 1.2.08: $^{13}$C NMR (75 MHz, CDCl$_3$) spectrum of compound 32
Chapter I Section B  
First Stereoselective Total synthesis of Rhoiptelol C

![FTIR Analysis Report](image1)

**Sample Name:** YV-RA-TOS [NEAT]
**Sample Preparation:**
**Collection time:** Fri Dec 2111 54:57 2012 (GMT-05:00)
**Bench:** Thermo Nicolet Nexus 670 Spectrometer
**Resolution:** 4cm-1

**Fig. 1.2.09:** IR spectrum of compound 32

![ESI-MS spectrum](image2)

**Fig. 1.2.10:** ESI-MS spectrum of compound 32
Chapter I Section B  First Stereoselective Total synthesis of Rhoiptelol C

Fig. 1.2.11: $^1$H NMR (300 MHz, CDCl$_3$) spectrum of compound 33

Fig. 1.2.12: $^{13}$C NMR (75 MHz, CDCl$_3$) spectrum of compound 33
Chapter I Section B  
First Stereoselective Total synthesis of Rhoiptelol C

Fig. 1.2.13: IR spectrum of compound 33

Fig. 1.2.14: ESI-MS spectrum of compound 33
Chapter I Section B  
First Stereoselective Total synthesis of Rhoiptelol C

Fig. 1.2.15: $^1$H NMR (300 MHz, CDCl$_3$) spectrum of compound 35

Fig. 1.2.16: $^{13}$C NMR (75 MHz, CDCl$_3$) spectrum of compound 35
Chapter I Section B  
First Stereoselective Total synthesis of Rhoiptelol C

Fig. 1.2.17: IR spectrum of compound 35

Fig. 1.2.18: ESI-MS spectrum of compound 35
Fig. 1.2.19: $^1$H NMR (300 MHz, CDCl$_3$) spectrum of compound 36

Fig. 1.2.20: $^{13}$C NMR (75 MHz, CDCl$_3$) spectrum of compound 36
Chapter I Section B  
First Stereoselective Total synthesis of Rhoiptelol C

![Fig. 1.2.21: IR spectrum of compound 36](image)

Sample Name: YVL-RA-DETOS [NEAT]  
Sample Preparation:  
Collection time: Wed Jun 12 12:26:28 2013 (GMT+08)  
Bench: Thermo Nicolet Nexus 670 Spectrometer  
Resolution: 4cm⁻¹  
Detection: DTGS KBr  
Beam splitter: KBr  
Source: IR  
Analyst Name:  

![Fig. 1.2.22: ESI-MS spectrum of compound 36](image)
Chapter I Section B  
First Stereoselective Total synthesis of Rhoiptelol C

Fig. 1.2.23: $^1$H NMR (300 MHz, Me$_2$CO-d$_6$) spectrum of compound 28

Fig. 1.2.24: $^{13}$C NMR (75 MHz, Me$_2$CO-d$_6$) spectrum of compound 28