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Research Article

SYNTHESIS OF RACEMIC *ANTI*-1,3-DIOLS: COMPLEMENTARITY BETWEEN BARLUENGA REDUCTION AND ALDOL-TISHCHENKO REACTION

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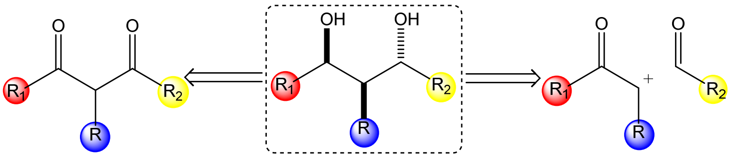
Abstract

Propionates are molecules with alternating hydroxyl and usually methyl groups along their carbon chain. The 1,3-diol motif is ubiquitous in most plant-derived molecules. These molecules generally possess remarkable biological properties. Given their importance, several synthetic methods exist, including the double reduction of diketones (Barluenga) and the Tischenko aldol condensation. By applying these two methods, fifteen diols were synthesized, including six C2-pseudo-symmetric diols by Barluenga reduction with a very good yield (60 to 77%) and very remarkable diasereoselectivities (dr= 75:25 to 98:02) and nine other molecules by Tischenko aldolization, including two pseudo-symmetric diols, with good diasereoselectivities (58:42 to 97:03) and moderate yields (14 to 68%). Diastereoselectivity is determined by integrating the proton spectrum of the unpurified product.



Scheme :1,3-Diol Retrosynthesis

This study demonstrates the complementarity and effectiveness of the twosynthesismethods.

Keywords:*Anti-1,3*-diols, Barluenga reduction, Aldol-Tishchenko reaction.

INTRODUCTION

The 1,3-diol motif represents a ubiquitous structural feature in many natural products, particularly among polyketides, as well as in bioactive agents and pharmaceuticals, making the development of efficient methods for their synthesis an important research objective [1]. Alcohols and diols are widely common units in natural molecules and play a key role in their biological activities. This importance has increased chemists' curiosity regarding diols, leading to considerable development of their synthetic methods to complement the total synthesis of large molecules [2]. Several methods exist for the synthesis of diols [3],[4],[5]. Despite the various methods used for the synthesis of these fragments, the aldol condensation/reduction route remains the most widely used. However, other methods exist, such as the aldol-Tishchenko reaction, the oxymercuration of cyclopropylcarbinols, and the photooxygenation of 1-methyl-2,3-diarylcyclopropanes followed by palladium-catalyzed hydrogenation on carbon (Pd/C). Asymmetric hydrogenation of dicarbonyls [6] and the catalyzed alkylation and allyllititanation of β-hydroxy aldehydes can also be mentioned. [3]. However, it should be noted that each method has limitations, but some of them can be complementary. In our case, the aim is to demonstrate the complementarity between the Barluenga double reduction of dicarbonyls [7] and the Tishchenko aldol condensation [8], [9]. In our study, we considered molecules containing aromatic fragments in their structures because these fragments play an important role in their stability. In particular, when used as ligands in organometallic catalysis, they can create π-π or π-π* interactions that lead to complex but highly stable and selective transition states.

RESULTS AND DISCUSSION

Double reduction of Barluenga

We first performed a conventional monoalkylation [10] of *β*-diketones in the presence of a base to attach various substituents to the C2 position of dibenzoylmethane, yielding, after reduction, molecules with three pro-stereogenic centers. This reaction was carried out in the presence of the inorganic base Na_2CO_3 and a halogenated derivative compound.

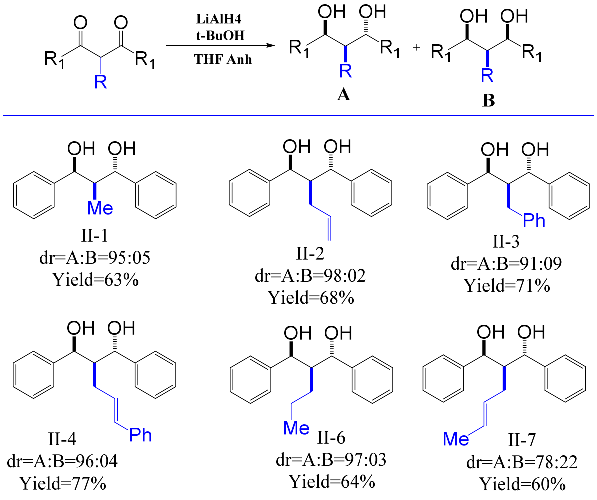


Figure 1. Scoop on the double reduction of *β*-diketones

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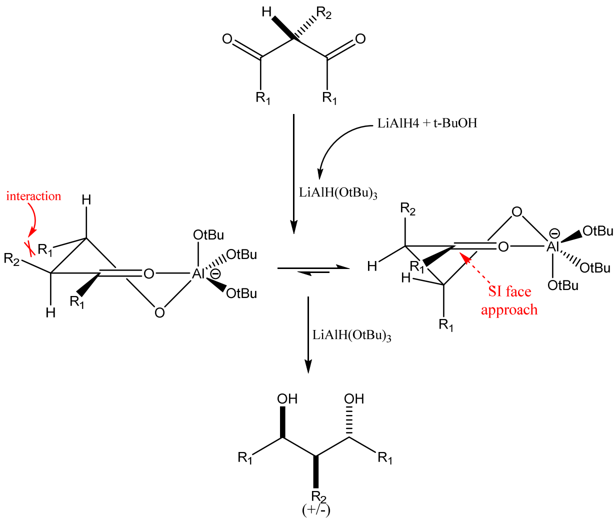
By applying the Barluenga method, we were able to successfully reduce our substrates, and the diols were obtained with very good selectivity for molecules II-1, II-2, II-3, II-4, II-5, and II-6. A decrease in the diasteroselectivity of diol II-7 (78:22 dr) was observed, which may be due to the coexistence of the two stereoisomers Z and E resulting from the small size of the methyl group. The yields are generally good.



Scheme 1 : Mechanism of the double reduction of β-diketones

The authors observed that the reduction was highly selective in favor of the α *(Syn/anti)* triad. They explain the diastereoselectivity of the second reduction by the preferential formation of a twisted boat conformer where groups R_1 and R_2 do not sterically interfere with each other. A second hydride would then approach via the Si face, opposite group R_2, to form the α *(syn/anti)* triad.

Aldol-Tishchenko reaction

We used different aldehydes and different ketones to obtain various molecules in order to determine the influence of the groups on the reaction.

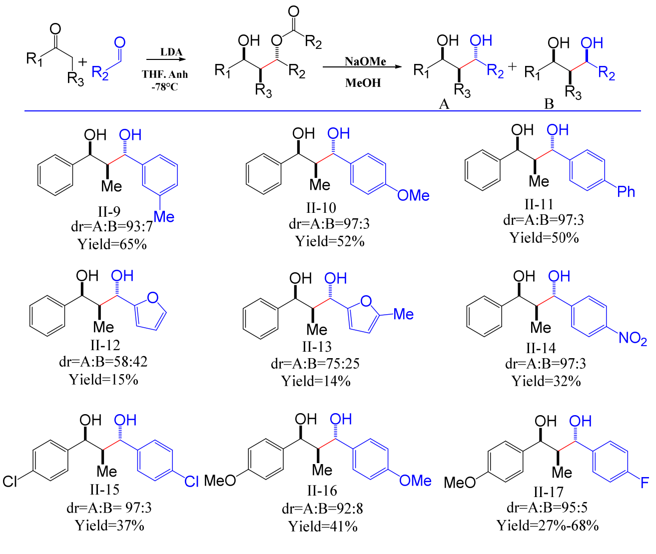
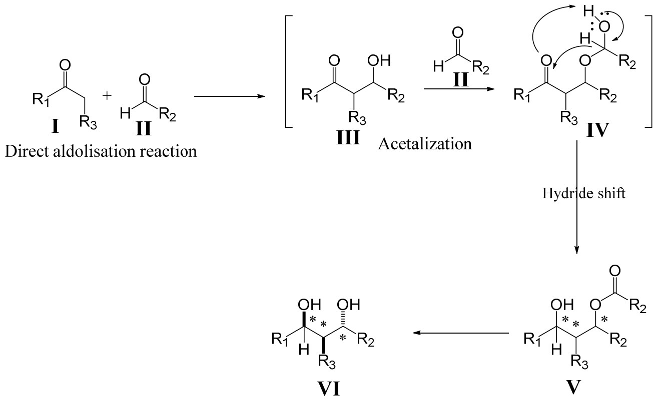


Figure 2. Scoop on Aldol-tishchenko's reaction

Inspired by the work of Bodnar and Marwal [6], we synthesized the above compounds with very good selectivities except in the cases where the aldehyde is a furfural derivative (II-12; 58:42 rd and II-13; 75:25 rd). The yields range from moderate to good, except for II-12 and II-13 where low yields were observed. These relatively low yields were obtained by Bodnar and his collaborators during the synthesis of diols and triols via an aldol-tishchenko tandem reaction [9]. They demonstrated that the yields for the *β*-hydroxy esters resulting from the first step are relatively high and that a decrease in yield is observed after hydrolysis of the monoester without any change in diastereoselectivity. A methyl group on the furfural ring improves the selectivity. By applying this method, we synthesized various C2 pseudo-symmetric and asymmetric molecules. The reaction tolerates both electron-withdrawing and electron-donating substituents on the aromatic ring. A heteroaromatic ring such as furfural leads to decreased selectivity and a slowing of the reaction. The advantage of this method is that it is carried out without intermediate steps; in other words, no product is isolated after the first step, and saponification proceeds directly to access the *anti* 1,3-diol. In our work, lithium diisopropylamine (LDA) was prepared in situ by mixing diisopropylamine and *n*-butyl lithium at 0°C.



Scheme 2. Mechanism of the aldol-tishchenko reaction

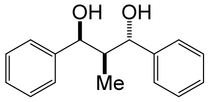
As shown in Scheme 2, a first equivalent of ketone **I** reacts with an equivalent of aldehyde **II** by direct adolazation, β-hydroxyketone **III** is formed which then reacts with a second equivalent of aldehyde **II** by acetalization to form an acetal **IV**, then there is an intramolecular hydride transfer leading to the diastereoselective reduction of the ketone, the β-hydroxyester **V** is obtained which will be saponified to obtain the β-diol in anti. **VII** [11]. The yields of products obtained with the Barluenga double reduction are higher than those obtained with the Tishchenko aldol condensation, which could be explained by the experimental conditions. Indeed, the Tischenko aldol condensation is carried out at a temperature of -78°C and with a specific order of successive addition of reactants; their reaction time is twice that of the double reduction.

Conclusion

Fifteen diols were synthesized, including six C2-pseudosymmetric diols produced by Barluenga reduction with very good yields and remarkable diastereoselectivities, and nine more, including two pseudosymmetric diols, with good diastereoselectivities and moderate yields. This study demonstrates the complementarity and efficiency of the two synthetic methods. The complementary nature of the two methods is clearly justified by the diversity of the synthesized molecules and their starting reagents. A possible study could be done on the double splitting of 1,3-diols.

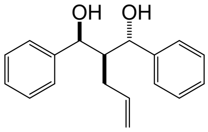
General procedure for the synthesis of 2-substituted 1,3-diphenylpropane-1,3-diols (Double reduction of diketone)

To a suspension of $LiAlH_4$ (4.0 mmol), in anhydrous THF (3 mL), t-BuOH (12.0 mmol) was added at 0°C. The evolution of hydrogen gas was monitored. When gas evolution ceased, diketone (1.0 mmol) in dry THF (2 mL) was added to the reaction mixture at the same temperature. After the addition was complete, it was slowly warmed to room temperature and stirred for 6 h. the reaction was followed by TLC. Once the reaction was complete, a saturated solution of sodium tartrate was added slowly to the reaction mixture at 0° C. and then stirred for 10 minutes at room temperature. It was then filtered through a layer of celite, washed with CH_2C1_2. The organic layer was separated and the aqueous layer was extracted with CH_2Cl_2. The combined organic layer was washed with brine, dried ($MgSO_4$) and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel (petroleum ether:ethyl acetate, 80:20) to give pure product.

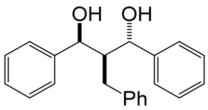


^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.44 – 7.25 (m, 10H), 5.08 (s, 1H), 4.75 (d, *J* = 7.0 Hz, 1H), 3.03 (s, 1H), 2.97 (s, 1H), 2.23 (td, *J* = 7.0, 2.5 Hz, 1H), 0.79 (d, *J* = 7.1 Hz, 3H).
^{13}C NMR (CDC13) 143.49, 142.62,129.32-126.12, 77.41, 73.99,45.65,11.09
HRMS, m/e 242 (M+),224, 118, 117, 77. Anal. Caled for $C_{16}H_{18}0_2$: C, 79.33; H, 7.43. Found: C, 79.45; H, 7.45.

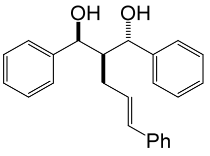
NMR according to the littérature [7]
mp=95°C



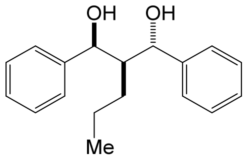
^{1}H NMR (300 MHz, Chloroform-*d*) δ 7.29 – 7.20 (m, 4H), 7.18 – 7.11 (m, 3H), 7.10 – 7.02 (m, 3H), 5.55 (dddd, *J* = 17.0, 10.1, 8.0, 6.0 Hz, 1H), 4.95 – 4.79 (m, 4H), 3.20 (d, *J* = 3.0 Hz, 1H), 2.96 (d, *J* = 4.8 Hz, 1H), 2.25 – 2.10 (m, 1H), 1.96 – 1.76 (m, 2H).
^{13}C NMR δC (125.8 MHz, CDCl 3 ; TMS) 28.96,51.09, 72.72,74.90, 116.89, 125.74, 125.80, 128.06, 128.51, 126.94, 127.40, 137.14, 142.52, 143.55.
HRMS [m/z (% rel.int.)]: 209.09 (23), 143.09 (26), 129.07 (100), 107.05 (28),77.04 (30); Calc. for C 5 H 7 O 107.04969, observed 107.04944.
NMR according to the littérature [12]
mp=119°C



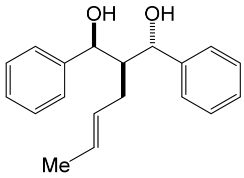
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.41 – 7.30 (m, 7H), 7.28 – 7.23 (m, 5H), 7.22 – 7.16 (m, 1H), 7.12 – 7.08 (m, 2H), 5.02 (t, *J* = 2.0 Hz, 1H), 4.91 – 4.81 (m, 1H), 3.35 (d, *J* = 2.4 Hz, 1H), 3.17 (d, *J* = 5.2 Hz, 1H), 3.03 – 2.57 (m, 2H), 2.27 (dtt, *J* = 10.2, 3.9, 1.9 Hz, 1H).
^{13}C NMR ($CDCl_3$) 143.56, 142.77,142.56,128.83-125.35, 73.71, 72.36, 53.36, 30.00
HRMS, m/e 318 (M+), 300, 282,194,179, 91. Anal. Caled for $C_{22}H_{22}0_2$: C, 83.01; 6.91. Found: C, 83.19; H, 6.95.
NMR according to the littérature [7]



^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.47 – 7.38 (m, 4H), 7.36 – 7.30 (m, 3H), 7.29 – 7.27 (m, 5H), 7.27 – 7.23 (m, 2H), 7.19 (ddd, *J* = 8.7, 4.9, 3.9 Hz, 1H), 6.39 (dt, *J* = 15.6, 1.3 Hz, 1H), 6.07 (ddd, *J* = 15.7, 8.1, 6.1 Hz, 1H), 5.06 (dt, *J* = 5.5, 3.1 Hz, 2H), 3.48 (d, *J* = 2.9 Hz, 1H), 3.38 (d, *J* = 4.9 Hz, 1H), 2.55 – 2.43 (m, 1H), 2.16 – 2.09 (m, 2H).
^{13}C NMR (75 MHz, Chloroform-*d*) δ 143.69, 142.56, 137.68, 132.00, 129.06, 128.54, 128.15, 127.36, 127.05, 126.12, 125.91, 75.02, 72.93, 51.60, 28.35 .
HRMS (ESI TOF) calculated for $C_{24}H_{24}O_2Na^+[M+Na]^+$ 367,1669 found 367,1666



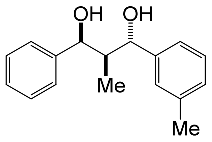
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.36 – 7.29 (m, 4H), 7.25 – 7.18 (m, 3H), 7.16 – 7.09 (m, 3H), 4.91 – 4.84 (m, 2H), 3.17 (dd, *J* = 4.2, 3.0 Hz, 2H), 1.90 (dtd, *J* = 9.2, 4.7, 2.1 Hz, 1H), 1.47 – 1.23 (m, 2H), 1.20 – 0.98 (m, 2H), 0.69 (t, *J* = 7.2 Hz, 3H).
^{13}C NMR (75 MHz, Chloroform-*d*) δ 143.89, 142.80, 128.42, 127.96, 127.25, 126.77, 125.77 (d, *J* = 11.6 Hz), 75.45, 73.17, 50.68, 26.26, 20.69, 14.04.
HRMS (ESI TOF) calculated for $C_{18}H_{22}O_2Na^+[M+Na]^+$ 293,1512 found 293,1513
mp= 90°C



Le diol **II-6** a été obtenu selon la procédure générale, à partir de dicétone **I-7** correspondante (7,5 mmol, 598 mg). Le produit brut a été purifié par chromatographie flash sur colonne de gel de silice en utilisant un mélange d'éther de pétrole et d'acétate d'éthyle (PE : EtOAc, 80 :20) pour donner le diol avec un rendement de 60 % sous la forme d'un solide blanc (1,28 g, 78 :22 rd).

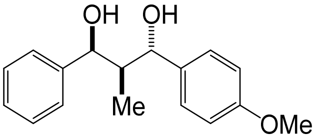
General Procedure for *Anti-1,3*-diols Synthesis :Aldol-Tishencko reaction

In freshly distilled diisopropylamine (1.1 mL, 7.9 mmol) was added to 20 mL of anhydrous THF. To the cooled (0°C) solution was added 2.5 M n-BuLi in hexanes (3.9 mL, 7.8 mmol). After 10 minutes the reaction mixture was cooled to -78°C. To the cooled reaction mixture was added ketone (1 mL, 7.8 mmol). After 20 minutes, aldehyde (2 mL, 17 mmol) was added dropwise (1 minute) and the resulting solution was stirred for 2 hours at -78°C. The reaction mixture was then warmed to 22°C, stirred for 12 h, quenched with 25 mL of saturated aqueous $NaHCO_3$, extracted with CH_2Cl_2, filtered through cotton wool and concentrated in vacuo. The residue was dissolved in MeOH (15 mL) and treated with NaOMe (mmol, 11 mol%) in MeOH (0.5 M, 0.1 mL). After 3 h the mixture was diluted with EtOAc (20 mL) and washed with H_20 (10 mL). The aqueous layer was extracted with EtOAc (2 x 20 mL) and the combined organic layers were washed with brine (20 mL), dried (Na_2SO_4) and concentrated under reduced pressure. The residue was purified by silica gel column chromatography (petroleum ether-EtOAc, 8:2) to give the diol.

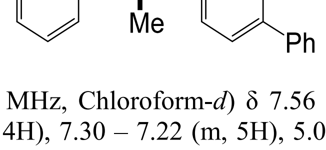


^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.37 – 7.31 (m, 3H), 7.30 – 7.25 (m, 3H), 7.18 – 7.11 (m, 2H), 5.05 (dd, *J* = 4.0, 2.4 Hz, 1H), 4.67 (dd, *J* = 7.0, 3.3 Hz, 1H), 3.41 (d, *J* = 4.1 Hz, 1H), 3.25 (d, *J* = 3.8 Hz, 1H), 2.39 (s, 3H), 2.26 – 2.15 (m, 1H), 0.75 (d, *J* = 7.1 Hz, 3H).
^{13}C NMR (75 MHz, Chloroform-*d*) δ 143.69, 142.56, 137.68, 132.00, 129.06, 128.54, 128.15, 127.36, 127.05, 126.12, 125.91, 75.02, 72.93, 51.60, 28.35 .

HRMS (ESI TOF) calculated for $C_{17}H_{20}O_2Na^+[M+Na]^+$ 279,1356, found 279,1357

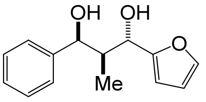


^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.37 – 7.26 (m, 7H), 6.93 – 6.88 (m, 2H), 5.05 (t, *J* = 3.1 Hz, 1H), 4.68 – 4.59 (m, 1H), 3.82 (s, 3H), 3.47 (d, *J* = 4.1 Hz, 1H), 3.22 (d, *J* = 3.7 Hz, 1H), 2.22 – 2.13 (m, 1H), 0.71 (d, *J* = 7.1 Hz, 3H).
^{13}C NMR (101 MHz, Chloroform-*d*) δ 158.94, 142.67, 135.76, 128.75 – 125.48 (m,9C,Ar), 113.78, 74.51, 55.27, 45.79, 11.46
.
HRMS (ESI): calcd. for $C_{17}H_{20}O_3$ Na [M + Na] + 295.1305; found : 295.1293
mp=72°C

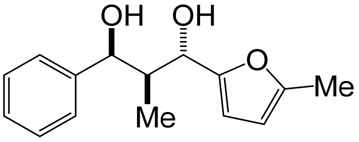


^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.56 – 7.51 (m, 5H), 7.40 – 7.35 (m, 4H), 7.30 – 7.22 (m, 5H), 5.02 (t, *J* = 2.7 Hz, 1H), 4.69 (dt, *J* = 8.3, 4.2 Hz, 1H), 2.97 (t, *J* = 3.6 Hz, 2H), 2.17 (pd, *J* = 7.1, 2.5 Hz, 1H), 0.73 (d, *J* = 7.1 Hz, 3H).
^{13}C NMR (101 MHz, $CDCl_3$) δ = 142.7, 142.6, 140.8, 140.5, 128.8, 128.1, 127.3, 127.2, 127.1, 127.0, 126.7, 126.0, 77.6, 74.4, 45.9, 11.3.

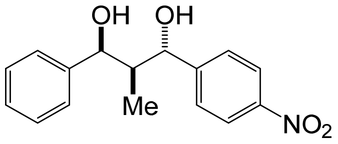
HRMS (ESI TOF) calculated for $C_{22}H_{22}O_2Na^+[M+Na]^+$ 341,1515, found 341,1512
mp=142°C



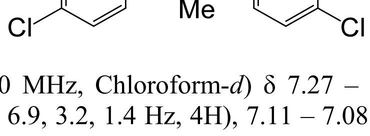
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.43 (dd, *J* = 1.8, 0.9 Hz, 1H), 7.40 – 7.34 (m, 4H), 7.29 (d, *J* = 1.3 Hz, 1H), 6.38 (dd, *J* = 3.2, 1.8 Hz, 1H), 6.34 (dt, *J* = 3.2, 0.8 Hz, 1H), 5.18 – 5.10 (m, 1H), 4.75 (dd, *J* = 7.0, 4.8 Hz, 1H), 3.08 (d, *J* = 5.0 Hz, 1H), 2.76 (d, *J* = 4.0 Hz, 1H), 2.43 (pd, *J* = 7.1, 2.6 Hz, 1H), 0.79 (d, *J* = 7.1 Hz, 3H).
^{13}C NMR (101 MHz, Chloroform-*d*) δ 155.96, 142.27 (d, *J* = 54.6 Hz,2C,Ar), 129.02 – 124.89 (m,5C,Ar), 110.20, 106.97, 74.39, 71.56, 43.57, 10.42
mp= 110°C



^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.37 (d, *J* = 4.4 Hz, 4H), 7.32 – 7.26 (m, 1H), 6.18 (d, *J* = 3.1 Hz, 1H), 5.94 (dq, *J* = 3.1, 1.0 Hz, 1H), 5.16 (d, *J* = 2.7 Hz, 1H), 4.65 (d, *J* = 7.3 Hz, 1H), 3.00 (d, *J* = 20.2 Hz, 2H), 2.41 (pd, *J* = 7.1, 2.7 Hz, 1H), 2.32 (d, *J* = 1.0 Hz, 3H), 0.76 (d, *J* = 7.1 Hz, 3H).
^{13}C NMR (75 MHz, Chloroform-*d*) δ 153.94, 151.73, 142.74 (d, *J* = 13.5 Hz,2C,Ar), 129.32 – 124.80 (m, 4C,Ar), 107.90, 106.04, 74.30 (1C-OH), 71.31 (1C-OH), 43.48, 13.85, 10.64 .
HRMS (ESI TOF) calculated for $C_{15}H_{18}O_3Na^+[M+Na]^+$ 269,1148, found 269,1147

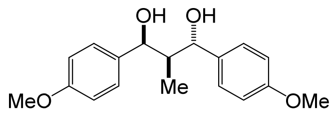


^{1}H NMR (400 MHz, Chloroform-*d*) δ 8.27 – 8.24 (m, 2H), 7.61 – 7.54 (m, 2H), 7.39 – 7.34 (m, 2H), 7.32 – 7.26 (m, 3H), 4.99 (t, *J* = 2.8 Hz, 1H), 4.85 (d, *J* = 1.7 Hz, 1H), 3.82 (d, *J* = 4.8 Hz, 1H), 2.68 (d, *J* = 3.3 Hz, 1H), 2.22 (pd, *J* = 7.1, 2.6 Hz, 1H), 0.83 (d, *J* = 7.1 Hz, 3H).
^{13}C NMR (101 MHz, Chloroform-*d*) δ 151.25, 147.73 (1C-NO_2), 141.84, 128.74 – 123.09 (m,9C,Ar)), 74.65 (1C-OH), 64.01 (1C-OH), 45.67, 11.23.
mp= 102°C



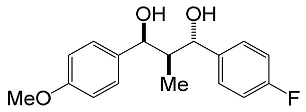
^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.27 – 7.23 (m, 2H), 7.20 (ddd, *J* = 6.9, 3.2, 1.4 Hz, 4H), 7.11 – 7.08 (m, 2H), 4.85 (t, *J* = 3.0 Hz, 1H), 4.59 – 4.55 (m, 1H), 3.27 (dd, *J* = 12.2, 3.9 Hz, 2H), 2.00 (qd, *J* = 7.0, 2.5 Hz, 1H), 0.64 (d, *J* = 7.1 Hz, 3H).
^{13}C NMR (75 MHz, Chloroform-*d*) δ 141.80, 140.84, 133.36, 132.78, 128.65, 128.23, 127.57, 127.36, 77.11, 73.77, 45.67, 11.25 .

HRMS (ESI TOF) calculated for $C_{16}H_{16}Cl_2O_2Na^+[M+Na]^+$ 333,0420 found 333,0416
mp= 71°C



^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.30 – 7.21 (m, 4H), 6.92 – 6.86 (m, 4H), 5.02 – 4.95 (m, 1H), 4.60 (dd, *J* = 7.4, 2.9 Hz, 1H), 3.82 (d, *J* = 1.1 Hz, 6H), 3.39 (d, *J* = 4.3 Hz, 1H), 3.24 (d, *J* = 3.4 Hz, 1H), 2.16 (pd, *J* = 7.2, 2.7 Hz, 1H), 0.70 (d, *J* = 7.2 Hz, 3H).
^{13}C NMR (75 MHz, Chloroform-*d*) δ 158.97, 158.57, 135.75, 134.61, 127.44 (d, *J* = 16.0 Hz), 113.58 (d, *J* = 27.4 Hz), 77.21, 74.67, 55.26 (d, *J* = 1.2 Hz), 45.74, 11.74

HRMS (20 eV) m/z 302 (M'). Anal. Calcd for $C_{18}H_{22}0_4$: C, 71.50; H, 7.33. Found: C, 71.51; H, 7.40.
mp=102°C



^{1}H NMR (400 MHz, Chloroform-*d*) δ 7.27 – 7.21 (m, 2H), 7.19 – 7.10 (m, 2H), 6.99 – 6.92 (m, 2H), 6.82 – 6.77 (m, 2H), 4.87 (t, *J* = 3.0 Hz, 1H), 4.55 (dd, *J* = 7.2, 2.9 Hz, 1H), 3.73 (s, 3H), 3.38 (d, *J* = 3.7 Hz, 1H), 3.02 (d, *J* = 3.8 Hz, 1H), 2.05 (dtd, *J* = 14.3, 7.1, 2.9 Hz, 1H), 0.62 (d, *J* = 7.1 Hz, 3H).
^{13}C NMR (101 MHz, Chloroform-*d*) δ 158.58, 139.41 (d, *J* = 3.1 Hz, 2C), 134.30, 129.06– 126.61 (m,4C), 116.25– 112.14 (m,4C), 76,80 (1C-OH),74.61 (1C-OH), 55.21, 45.60, 11.76 .
HRMS (ESI TOF) calculated for $C_{17}H_{19}FO_3Na^+$[M+Na]$^+$ 313,1210, found 313,1215

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