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Synthesis of α-hydroxy, β-bromo-*exo*-dicyclopentadiene-1-one: A new building block for cyclopentanoids

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Herein, we described a simple synthetic strategy for the preparation of α-hydroxy, β-bromo-*exo*-dicyclopentadiene-1-one by following a three-step sequence. The synthetic strategy relies on operationally simple reactions and high yielding steps. The newly synthesized compounds have been characterized by NMR. As the disclosed compound α-hydroxy, β-bromo-*exo*-dicyclopentadiene-1-one is highly functionalized but structurally similar to *exo*-dicyclopentadiene-1-one and it can be used a key building block for expanding the chemical space of cyclopentanoid derivatives.

Keywords: Bromination, Cyclopentanoids, Dicyclopentadiene-1-one, Epoxidation, Norbornene

Bridged-bicyclic compounds are useful candidates in organic synthesis and they have several applications in chemical science such as pharmaceutical chemistry, polymer chemistry, and material chemistry[1]. Besides, bridged-bicyclic compounds with ring-strain were found to be handy in natural product synthesis and exhibits useful biological properties[2]. Hence, there is a considerable amount of research activity in this regard.

Dicyclopentadiene (DCPD) is a unique example of bridged-bicyclic compounds and it exits in both *endo* and *exo* forms[3]. Though the *endo*-DCPD **1** is commercially available, the *exo*-isomer **2** can be synthesized by following two-step synthetic sequence. Due to ready access to *endo*-DCPD derivatives **3-4**, they are used as a starting materials (SMs) in synthetic chemistry. For example, *endo*-DCPD-1-one **4** was used to create useful compounds such as oxa-cages, intricate polycyclic compounds, and polyquinanes, *etc*. Whilst, there is a lot more to explore with *exo*-DCPD **2** and its derivatives **5-10**, especially in the metathesis area. The *exo*-DCPD derivatives shows enhanced reactivity towards olefin metathesis as compared to *endo*-DCPD derivatives[4].

We previously reported an improved and alternative synthetic pathway to *exo*-DCPD **2** and its enone derivative, *exo*-DCPD-1-one **7**. During this process we also realized useful intermediates **5-10** for synthetic chemistry (Fig. 1)[5].

Next, we have also prepared various precursors from *exo*-DCPD-1-one **4** and *endo*-DCPD-1-one **9** and used them to construct various polyquinane derivatives by using olefin metathesis as a key step (Fig. 2)[6].

Results and Discussion

Inspired by above interesting results of the building block **7** in polyquinanes synthesis, we aim to prepare analogues compounds of *exo*-enone **7**. The resulting analogues compounds can serve as key SMs in

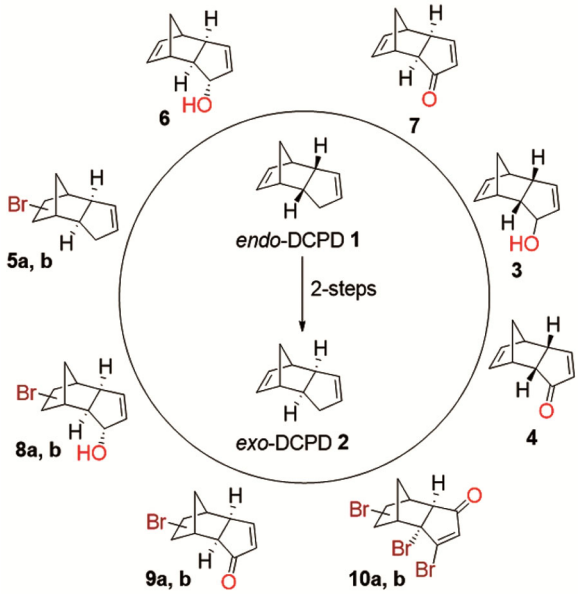


Fig. 1 — Various derivatives of *endo*- and *exo*-DCPD

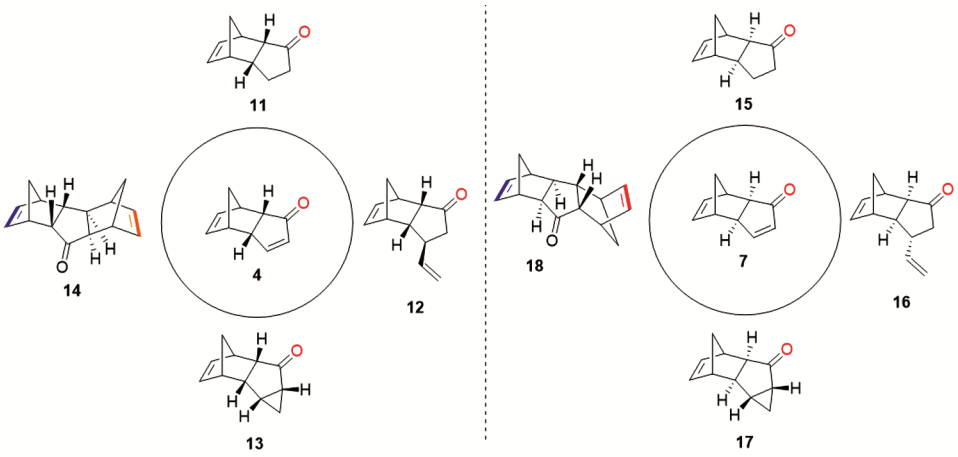
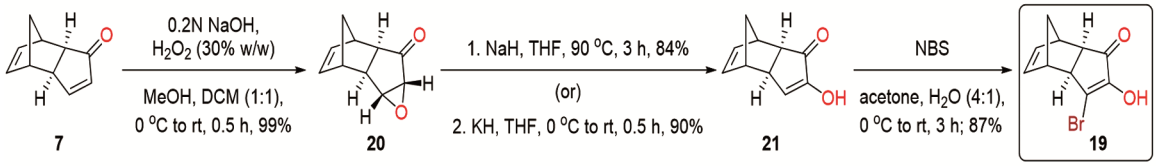


Fig. 2 — Various derivatives of *endo*- and *exo*-DCPD-1-one



Scheme 1 — Synthesis of heteroatoms (O, N, Br) containing *exo*-DCPD derivative **19**

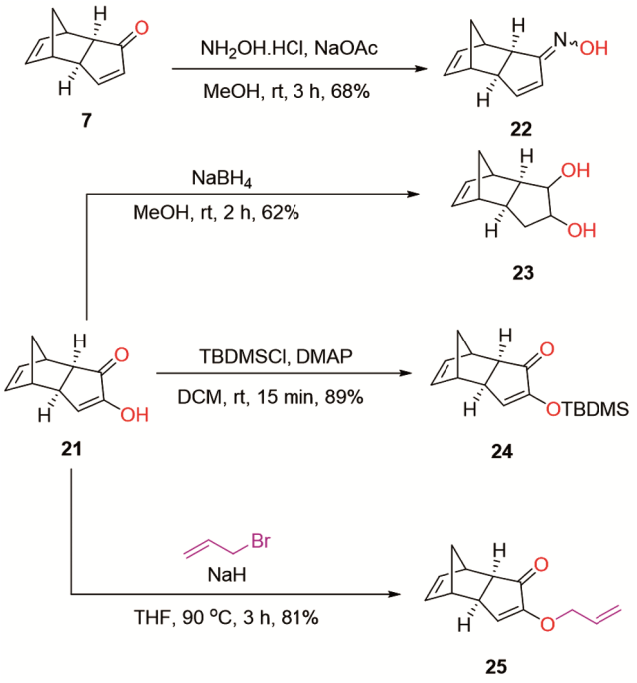
organic synthesis to expand the chemistry of *exo*-DCPD **2**. In this context, we identified highly functionalized *exo*-DCPD derivative **19** starting with *exo*-enone **7** (Scheme 1)[5]. For this purpose, *exo*-enone **7** was subjected to selective epoxidation to deliver the corresponding epoxy derivative **20** (Ref. 7). Then, this compound was treated with NaH or KH to afford a oxirane-ring opening compound, α-hydroxy enone derivative **21**. Finally, compound **21** was subjected to bromination under NBS reaction conditions to furnish a α-hydroxy-β-bromo enone derivative **19** (Ref. 8). Here, we believe that the highly functionalised compound **19** can serve as a key starting material for various chemical transformations. For example, compound **19** is useful for 1,2-addition, 1,4-addition, epoxidation, cycloadditions, *C*-alkylation, *O*-alkylation, various coupling reactions, *etc*. as it contains different functional groups (enone, –OH, –Br, and strained norbornene bond).

After successful synthesis of compound **19**, the key intermediates are used for the derivatization and realized useful precursors (Scheme 2). When compound **7** was treated with $NH_2OH.HCl$, oxime **22** was obtained in 68% yield. The 2-hydroxy enone **21** underwent reduction with $NaBH_4$ and produced 1,2-hydroxy derivative **23** in 62% yield. Additionally, the compound **21** was treated with TBDMS-Cl to obtain the *O*-TBDMS protected compound **24** in 89% yield. Along similar lines, hydroxy compound **21** underwent allylation to deliver *O*-allylated compound **25** in 81% yield.

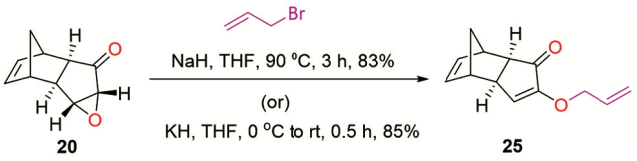
Interestingly, *O*-allylated compound **25** realized from **20** in one-pot reaction involving oxirane ring open followed by allylation in the presence of NaH or KH and allyl bromide (Scheme 3).

Experimental Section

All of the chemical solvents were purchased from Merck company and used without further purification. All the air-and moisture sensitive reactions were carried out in anhydrous and/or degassed solvents using an oven-dried and desiccator-cooled multi-neck round-bottom flask (RBF) fitted with rubber septa and/or glass stopper under nitrogen (N_2) atmosphere (atm). The reaction progress was monitored by thin-layer chromatography (TLC) using the appropriate mobile phase (ethyl acetate (EtOAc)/pet ether (PE)) and UV-inactive spots are visualized by using TLC stain solutions such as I_2, $KMnO_4$, and PMA. NMR spectra of all the synthesized compounds were obtained by using Bruker (AVANCE IIITM) 500 MHz and Bruker (AVANCE IIITM/AscendTM) 400 MHz spectrometers and solvent residual peaks as an internal standard (^{1}H NMR: $CDCl_3$ at 7.26 ppm; $^{13}C\{^1H\}$ NMR: $CDCl_3$ at 77.2 ppm). ^{1}H NMR data expressed in chemical shift (δ ppm), multiplicity (s, singlet; bs, broad singlet; d, doublet;



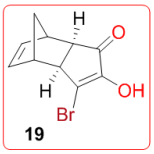
Scheme 2 — Synthesis of key intermediates **22-25**



Scheme 3 — One-pot synthetic route to compound **26**

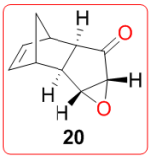
t, triplet; q, quartet; quint, quintet; m, multiplet; dd, doublet of doublet; ddd, doublet of doublet of doublet; dt, doublet of triplet; td, triplet of doublet, *etc*.), and coupling constants (*J* in Hertz; Hz). High-resolution mass spectrometry (HRMS) measurements of unknown compounds were done by using Bruker (Maxis Impact)/Micromass Q-ToF spectrometers and Agilent (AdvanceBio 6545XT LC/Q-TOF). Infrared (IR) spectra of all new compounds were collected from a Nicolet Impact-400 FTIR spectrometer.

Synthesis of 3-Bromo-2-Hydroxytricyclicenone 19


19

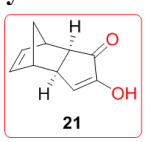
To a magnetically stirred solution of α-hydroxyenone **21** (200 mg, 1.233 mmol) in acetone/water (4:1; 10 mL) was added NBS (263 mg, 1.479 mmol) at 0°C and stirred at RT for 3 h. At the conclusion of the reaction (TLC monitoring), the RM was diluted with water and the solvent mixture was extracted with EtOAc (20–30 mL) thrice. The organic layer was concentrated and the crude product was purified by column chromatography (5–10% EtOAc/PE as an eluent) to afford **19** (258 mg, 87%) as a semi-solid. R_f 0.21 (10% EtOAc/PE mobile phase). ^{1}H NMR (500 MHz, $CDCl_3$): δ 6.56 (s, 1H), 6.29 (dd, *J* = 5.54, 3.15 Hz, 1H), 6.24 (dd, *J* = 5.51, 2.93 Hz, 1H), 2.96 (s, 1H), 2.91 (s, 1H), 2.81 (d, *J* = 4.89 Hz, 1H), 2.44 (d, *J* = 4.86 Hz, 1H), 1.47 (d, *J* = 9.72 Hz, 1H), 1.26 (d, *J* = 9.72 Hz, 1H); ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ 199.7 (CO), 153.6 (C), 137.8 (CH), 137.3 (CH), 131.2 (C), 52.3 (CH), 49.3 (CH), 43.3 (CH), 42.5 (CH), 41.3 (CH_2); IR (neat): 3282, 1693, 1651, 1368, 1155, 675 cm^{-1}.

Synthesis of *α*, β-epoxyketone 20


20

To a vigorously stirred solution of enone **7** (3.6 g) in MeOH/DCM (1:1; 60 mL) was added 0.2N aq. NaOH solution (14.5 mL) followed by H_2O_2 solution (30%w/w in H_2O; 14.5 mL) at 0°C and stirred at RT 0.5 h. At the conclusion of the reaction (by TLC monitoring), RM was diluted with water and the solvent mixture was extracted with DCM (100 mL) thrice. The organic layer was concentrated and the crude product was purified by silica gel column chromatography (2–5% EtOAc/PE as an eluent) to afford **20** (3.5 g, 88%) as a colorless liquid. R_f 0.55 (10% EtOAc/PE mobile phase). ^{1}H NMR (400 MHz, $CDCl_3$): δ 6.27 (dd, *J* = 5.58, 3.24 Hz, 1H), 6.20 (dd, *J* = 5.59, 2.59 Hz, 1H), 3.70–3.69 (m, 1H), 3.53–3.52 (m, 1H), 3.04 (s, 1H), 2.96 (s, 1H), 2.47 (d, *J* = 6.22 Hz, 1H), 2.15–2.13 (m, 1H), 1.40–1.36 (m, 1H), 1.28 (d, *J* = 9.43 Hz, 1H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 209.7 (CO), 139.1 (CH), 137.8 (CH), 60.7 (CH), 59.4 (CH), 50.2 (CH), 47.5 (CH), 45.9 (CH), 43.7 (CH), 43.6 (CH_2).

Synthesis of 2-Hydroxytricyclicenone 21


21

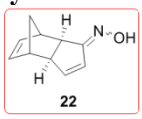
Method-A: To a stirred suspension of activated NaH (1.8 g, 35.141 mmol) in THF (20 mL) was added

a solution of epoxyketone **20** (1.9 g, 11.713 mmol) in THF (10 mL) at RT and heated to reflux at 90°C for 3 h. At the conclusion of the reaction (by TLC monitoring), RM was diluted with water and the solvent mixture was extracted with EtOAc (100 mL) thrice. The organic layer was concentrated and the crude product was purified by silica gel column chromatography (10–15% EtOAc/PE as an eluent) to afford **21** (1.6 g, 84%) as an semi-solid. R_f 0.37 (20% EtOAc/PE mobile phase).

Method-B: To a stirred suspension of activated KH (371 mg, 9.247 mmol) in THF (10 mL) was added a solution of epoxyketone **20** (500 mg, 3.082 mmol) in THF (10 mL) at RT and heated to reflux at 90°C. After 10 min, RM turned into brick red colour and after another 20 min, RM turned into ash colour or off-white colour. At the conclusion of the reaction (by TLC monitoring), RM was diluted with water and the solvent mixture was extracted with EtOAc (30–40 mL) thrice. The organic layer was concentrated and the crude product was purified by column chromatography (10–15% EtOAc/PE as an eluent) to afford **21** (450 mg, 90%).

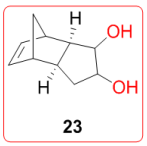
^{1}H NMR (400 MHz, $CDCl_3$): δ 6.48 (d, J = 2.99 Hz, 1H), 6.29 (dd, J = 5.51, 3.10 Hz, 1H), 6.18 (dd, J = 5.52, 2.96 Hz, 1H), 5.98 (bs, 1H), 2.88 (s, 1H), 2.63–2.61 (m, 2H), 2.25 (d, J = 4.62 Hz, 1H), 1.39 (d, J = 9.46 Hz, 1H), 1.27 (d, J = 9.44 Hz, 1H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 204.9 (CO), 155.9 (CH), 138.7 (CH), 136.5 (CH), 132.2 (CH), 50.0 (CH), 43.13 (CH), 43.12 (CH), 42.9 (CH), 41.1 (CH_2); IR (neat): 3344, 3060, 2974, 1694, 1393, 1208, 1098, 849, 832, 762, 709 cm^{-1}.

Synthesis of 1-Hydroxylaminetricyclic Compound 22



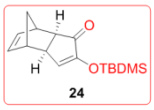
To a magnetically stirred solution of enone **7** (100 mg, 0.684 mmol) in MeOH (2.5 mL) was added $NH_2OH.HCl$ (71 mg, 1.026 mmol) followed by NaOAc (84 mg, 1.026 mmol) at RT and stirred for 1 h. At the conclusion of the reaction (by TLC monitoring), RM was concentrated and the crude product was purified by column chromatography (15–20% EtOAc/PE as an eluent) to afford **22** as an off-white semi-solid (75 mg, 68%). R_f 0.32 (10% EtOAc/PE mobile phase). ^{1}H NMR (400 MHz, $CDCl_3$): δ 8.98 (bs, 2OH), 6.79 (dd, J = 5.88, 1.51 Hz, 1H), 6.49 (dd, J = 5.79, 2.56 Hz, 1H), 6.43 (dd, J = 5.76, 2.57 Hz, 1H), 6.27 (dd, J = 5.76, 1.36 Hz, 1H), 6.24–6.18 (m, 3H), 6.14 (dd, J = 5.58, 2.95 Hz, 1H), 3.28 (s, 1H), 2.90 (d, J = 5.63 Hz, 1H), 2.84–2.78 (m, 3H), 2.70–2.64 (m, 3H), 1.39–1.36 (m, 3H), 1.33–1.30 (m, 1H); ^{13}C{^{1}H} NMR (100 MHz, $CDCl_3$): δ 167.6, 165.4, 149.7, 147.7, 138.3, 137.7, 137.6, 137.2, 132.2, 127.2, 52.4, 52.0, 46.5, 46.3, 45.1, 44.5, 43.5, 42.7, 41.5, 41.4.

Synthesis of 1,2-di-Hydroxytricyclic Compound 23



To a magnetically stirred solution of hydroxyenone **21** (100 mg, 0.616 mmol) in MeOH (5 mL) was added $NaBH_4$ (233 mg, 6.160 mmol) at 0°C and stirred at RT 2 h. At the conclusion of the reaction (by TLC monitoring), RM was diluted with water and the solvent mixture was extracted with EtOAc (20–30 mL) thrice. The organic layer was concentrated and the crude product was purified by column chromatography (15–20% EtOAc/PE as an eluent) to afford **23** (63 mg, 62%) as a semi-solid. R_f 0.35 (40% EtOAc/PE mobile phase). ^{1}H NMR (500 MHz, $CDCl_3$): δ 6.11–6.08 (m, 2H), 4.08–4.00 (m, 2H), 2.80 (s, 1H), 2.56 (s, 1H), 2.36 (bs, 2OH), 2.00 (dt, J = 14.11, 7.21 Hz, 1H), 1.85–1.76 (m, 3H), 1.34 (d, J = 8.71 Hz, 1H), 1.14 (q, J = 11.80 Hz, 1H); ^{13}C{^{1}H} NMR (125 MHz, $CDCl_3$): δ 138.7 (CH), 137.7 (CH), 76.8 (CH), 71.3 (CH), 46.3 (CH), 44.7 (CH_2), 44.0 (CH), 42.0 (CH), 40.6 (CH), 35.5 (CH_2).

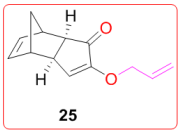
Synthesis of *O*-TBDMS Compound 24



To a magnetically stirred solution of hydroxyenone **21** (100 mg, 0.616 mmol) and imidazole (84 mg, 1.233 mmol) in DCM (5 mL) was added TBDMSCl (139 mg, 0.924 mmol) in one portion at RT. After 15 min, RM turned into white suspension at which reaction was completed (TLC monitoring). RM was diluted with water and the solvent mixture was extracted with DCM (20–30 mL) thrice. The organic layer was concentrated and the crude product was purified by column chromatography (2–3% EtOAc/PE as an eluent) to afford **24** (152 mg, 89%) as a pale brown semi-solid. R_f 0.90 (10% EtOAc/PE mobile phase). ^{1}H NMR (500 MHz, $CDCl_3$): δ 6.52 (d, J = 3.02 Hz, 1H), 6.25 (dd, J =

5.55, 3.09 Hz, 1H), 6.16 (dd, *J* = 5.51, 2.94 Hz, 1H), 2.87 (s, 1H), 2.60 (s, 1H), 2.5 (t, *J* = 3.82 Hz, 1H), 2.13 (d, *J* = 4.93 Hz, 1H), 1.35 (d, *J* = 9.31 Hz, 1H), 1.27 (d, *J* = 9.33 Hz, 1H), 0.93 (s, 9H), 0.18 (d, *J* = 2.55 Hz, 6H); $^{13}C\{^{1}H\}$ NMR (125 MHz, $CDCl_3$): δ 205.1 (CO), 156.9 (CH), 139.5 (CH), 138.3 (CH), 136.5 (CH), 49.8 (CH), 43.4 (CH), 43.3 (CH), 42.1 (CH), 41.1 (CH_2), 25.7 (3×CH_3), 18.5 (C), -4.3 (CH_3), -4.4 (CH_3); IR (neat): 3015, 2947, 2860, 1711, 1617, 1461, 1333, 1286, 1250, 1234, 1217, 1111, 842, 784, 754 cm^{-1}.

Synthesis of *O*-Allyl Compound 25



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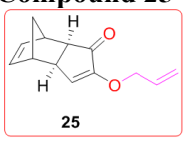
To a stirred suspension of NaH (3 equiv) in THF (10 mL) was added a solution of compound **21** (1 equiv) in THF (10 mL) and allyl bromide (2 equiv) at RT and heated to reflux at 90°C for 3 h. At the conclusion of the reaction (by TLC monitoring), RM was diluted with water and the solvent mixture was extracted with EtOAc (100 mL) thrice. The organic layer was concentrated and the crude product was purified by silica gel column chromatography (5–10% EtOAc/PE as an eluent) to afford **25** (403 mg, 81%) as a yellow liquid. R_f 0.52 (20% EtOAc/PE mobile phase).

^{1}H NMR (400 MHz, $CDCl_3$): δ 6.29–6.28 (m, 2H), 6.15 (dd, *J* = 5.52, 2.92 Hz, 1H), 5.99–5.90 (m, 1H), 5.35–5.24 (m, 2H), 4.41–4.39 (m, 2H), 2.89 (s, 1H), 2.60–2.57 (m, 2H), 2.19 (d, *J* = 4.86 Hz, 1H), 1.35 (d, *J* = 9.34 Hz, 1H), 1.27 (d, *J* = 9.34 Hz, 1H); $^{13}C\{^{1}H\}$ NMR (100 MHz, $CDCl_3$): δ 203.2 (CO), 159.0 (C), 138.4 (CH), 136.6 (CH), 132.2 (CH), 130.6 (CH), 118.6 (CH_2), 70.6 (CH_2), 50.4 (CH), 43.5 (CH), 43.4 (CH), 42.6 (CH), 41.1 (CH_2); IR: 3010, 2941, 1705, 1458, 1216, 759.

One Pot Synthetic Procedure to *O*-Allyl Compound 25



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Method-A: To a stirred suspension of NaH (393 mg, 9.864 mmol) in THF (10 mL) was added a solution of epoxyketone **20** (400 mg, 2.466 mmol) in THF (10 mL) and allyl bromide (2 equiv) at RT and heated to reflux at 90°C for 3 h. At the conclusion of the reaction (by TLC monitoring), RM was diluted with water and the solvent mixture was extracted with EtOAc (100 mL) thrice. The organic layer was concentrated and the crude product was purified by silica gel column chromatography (5–10% EtOAc/PE as an eluent) to afford **25** (413 mg, 83%).

Method-B: To a stirred suspension of KH (213 mg, 5.326 mmol) in THF (10 mL) was added a solution of epoxyketone **20** (144 mg, 0.887 mmol) in THF (10 mL) and allyl bromide (2 equiv) at RT and heated to reflux at 100°C for 0.5 h. At the conclusion of the reaction (by TLC monitoring), the RM was diluted with water and the solvent mixture was extracted with EtOAc (30–40 mL) thrice. The organic layer was concentrated and the resulting crude product was purified by column chromatography (2–5% EtOAc/PE as an eluent) to afford **25** (152 mg, 85%).

Conclusion

We have successfully synthesized α-hydroxy, β-bromo-*exo*-dicyclopentadiene-1-one **19** by following simple synthetic procedure involving three-step sequence. During the development of this synthetic strategy, the realized intermediates are shown for the further functionalization. To this end, a total of six new compounds (**19**, **21**, **22-25**) are reported in good yield and characterized by ^{1}H and ^{13}C NMR. Exploring the chemistry of these compounds in organic synthesis is a worthy exercise. Especially, the α-hydroxy, β-bromo-*exo*-dicyclopentadiene-1-one **19** is a useful intermediate to expand polyquinanes chemistry.

Supplementary Information

Supplementary information is available in the website http://nopr.niscpr.res.in/handle/123456789/58776.

Acknowledgement

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