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Reactions of 9,10-dioxatricyclo[6.2.2.0^{1,6}]dodeca-3,11-dien-6-yl hydroperoxide: synthesis of oxygenated naphthalene derivatives

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ABSTRACT

We synthesized 9,10-dioxatricyclo[6.2.2.0^{1,6}]dodeca-3,11-dien-6-yl hydroperoxide (**7**) and investigated for the first time its further chemical transformation with dimethylsulfide, NEt_3, PPh_3, and Co–TPP, respectively. A variety of stereospecifically oxygenated compounds were obtained from these reactions containing different functional groups such as alcohols, ketone, and epoxides.

1. Introduction

Polyhydroxycyclohexanes are of interest to those concerned with carbohydrates.[1] Carbohydrates are densely functionalized molecules and as a result of their synthetic application often require many reaction steps, including the manipulation of different protecting groups. Recently Mehta et al. developed a multistep procedure for the preparation of a new family of polyhydroxylated decalin **1** and annulated inositols **2**, from tetrahydronaphthalene. Our groups developed a method for synthesis of **1** from readily available tetrahydronaphthalene (Fig. 1).[2,3]

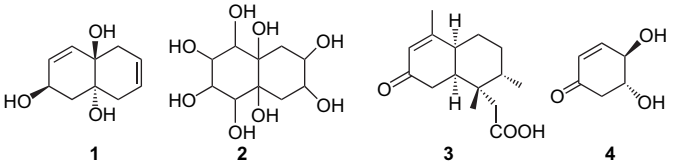


Figure 1.

There are also many different methods for the syntheses of functionalized *cis*-decalin ring systems due to the occurrence and isolation of many natural products **3**. Some of these natural products showed interesting biological activity.[2,3]

Optically active 4-hydroxycyclohex-2-en-1-one (**4**) has been employed in several laboratories as a starting material for the synthesis of many biologically active chiral α-hydroxy ketones.[3b]

The oxyfunctionalization of dienes and allylic systems in hydrocarbon subunit is efficiently performed by singlet oxygen.[4] It has been reported that the main reactions of singlet oxygen are cycloaddition and ene-reaction.[4]

While the 1,4-cycloaddition of singlet oxygen to cyclic diene results in the formation of bicyclic endoperoxide, the ene-reaction of singlet oxygen with olefins containing allylic hydrogens yields unsaturated hydroperoxides.[4]

The reaction of cyclic 1,4-diene systems with singlet oxygen rearranges the 1,3-diene unit along with hydroperoxide and this unit can be trapped by the singlet oxygen to form the endoperoxide[5,6] (Fig. 2).

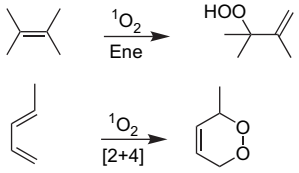
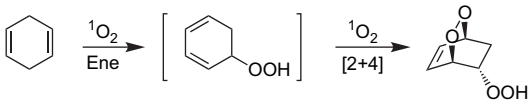


Figure 2.

Singlet oxygen addition to cyclic 1,3-diene systems is stereospecific and cleavage of endoperoxide by appropriate reducing reagents lead to cyclic 1,4-dihydroxyl compounds in a cis-configuration. So far, singlet oxygen methodology is unique in forming *cis*-1,4-diols in the literature (Scheme 1).[4]



Scheme 1.

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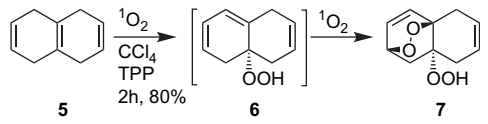
2. Results and discussion

The peroxide linkage is highly susceptible to reductive cleavage by a variety of reductants.[4] For example, the selective reductions of peroxide linkages have been performed with either thiourea, $LiAlH_4$, or dimethylsulfide under very mild conditions to give alcohols. The reaction of peroxides with NEt_3, PPh_3, and Co–TPP has been known to form hydroxyketones, mono-, and bis-epoxides, respectively.

Recently, we have developed a versatile synthetic approach,[3a,6] which is applicable to the synthesis of polyhydroxy tetrahydronaphthalene derivatives from the photooxygenation of isotetraline. The X-ray analysis confirmed that hydroperoxide and endoperoxide were anti to each other[3a] (Scheme 2).

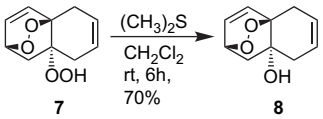


Scheme 2.

As seen, this method is a practical application for the synthesis of polyhydroxy hydrocarbon derivatives because three oxygen atoms have been incorporated into the molecule in one operation. To expand our recently reported method, we decided to develop a stereoselective route to tetrahydronaphthalene derivatives, which have hydroxyl, epoxy, and carbonyl group (**8**, **9**, **10**, **12**, **15**) as key compounds for synthesis of highly hydroxylated hydrocarbons and tetrahydronaphthalen-2-one derivatives.

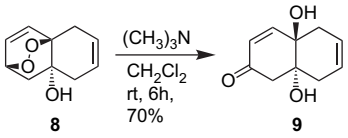
The starting material, the known tetrahydronaphthalene (**5**), was obtained from the metal–ammonia reduction[7] of naphthalene. Tetraphenylporphyrin (TPP) sensitized photooxygenation of tetrahydronaphthalene in methylene chloride at room temperature resulted in the formation of the endoperoxide **7** in an 80% yield (Scheme 2).[6]

There are two functional groups in compound **7**, endoperoxide (O–O) and hydroperoxide (–OOH). The hydroperoxide group in compound **7** has been selectively converted to –OH **8** by the reduction with dimethyl sulfide[8] at room temperature (Scheme 3). The structure of **8** was assigned by ^1H and ^{13}C NMR spectroscopy. In the ^1H NMR spectrum of compound **8**, there are five distinct AB-systems, which are four olefinic and six methylenic protons. Adjacent olefinic protons to two CH_2 resonates appear as broad singlet (as multiplet) at 5.67 ppm. Although the hydroperoxide proton of **7** resonated at 7.60 ppm, after the cleavage of hydroperoxide bond, the alcohol proton of **8** appeared at 2.00 ppm as a broad singlet. The double resonance spectrum and ^{13}C NMR spectrum are also fully consistent with this structure **8**.



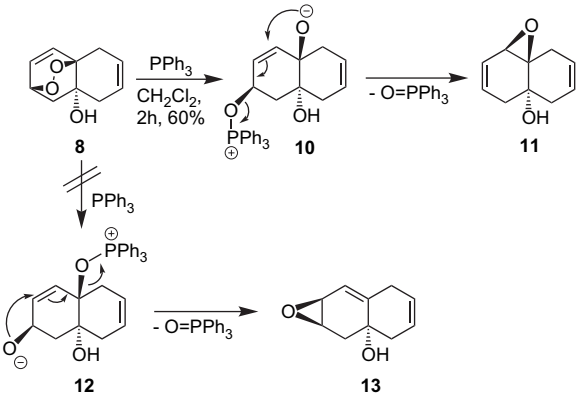
Scheme 3.

For the synthesis of fused bicyclic 4-hydroxy-2-cyclohexen-1-one derivative **9**, we used hydroxy-endoperoxide **8**. Treatment of hydroxy-endoperoxide **8** with NEt_3 in CH_2Cl_2 at room temperature gave **9** by a facile cleavage of the O–O linkage via Kornblum–DeLaMare rearrangement[9] (Scheme 4). Only one isomer was expected in this reaction because one of the endoperoxide carbons bears a hydrogen atom. The structure of **9** was assigned by ^1H and ^{13}C NMR spectroscopy. There are four distinct AB-systems in the ^1H NMR spectrum, which are two olefinic and six methylenic protons. Adjacent olefinic protons to two CH_2 and alcohol protons resonated at 5.93 and 4.84 ppm, respectively. Especially, double resonance experiments and 10 lines in the ^{13}C NMR spectrum are in good agreement with this structure.



Scheme 4.

Treatment of compound **8** with PPh_3 gave epoxy-alcohol as sole product[10] **11**. The formation mechanism of this epoxy-alcohol **11** is given in Scheme 5. Since the hydroxy-endoperoxide **8** has no plane of symmetry, PPh_3 can attack both oxygen atoms in the peroxide linkage to form the intermediates **10** and **12**. However, the epoxy-alcohol **11** can be formed only from the intermediate **10**.



Scheme 5.

Compounds hydroperoxide **7** and alcohol **8** contain the same general structure. Thus, the X-ray of structure[3a] **7** can inform us about the approach of PPh_3. The X-ray crystal structure indicates clearly that PPh_3 approaches the peroxide unit in **8** exclusively from the sterically less crowded face of the molecule (Fig. 3) and this leads to intermediate **10**. The structure of epoxide **11** was determined by the ^1H and ^{13}C NMR spectrum. There is only one epoxy proton bonded to one of the epoxy carbons in compound **11**, this proton resonated at 3.85 ppm. ^1H NMR data for compound **11** confirm this result. The tertiary carbon atom of the epoxide resonated at 55.5 ppm and the quaternary epoxide carbon resonated at 63.3 ppm in the ^{13}C NMR spectrum. All NMR spectral data are in agreement with this structure.

On the other hand, unsaturated bicyclic endoperoxides have proven extremely useful in synthesis as they are readily convertible

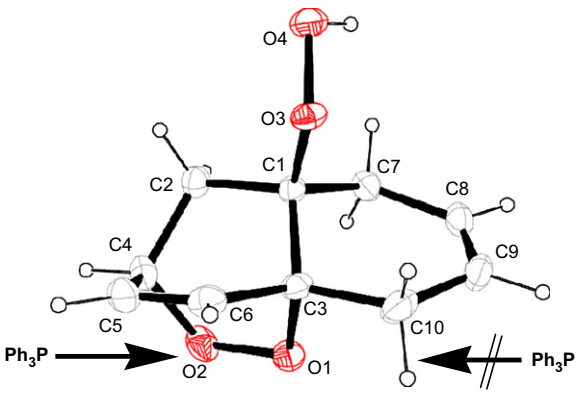
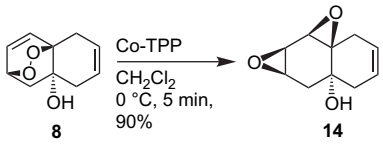


Figure 3. The X-ray structure of hydroperoxide **7**.

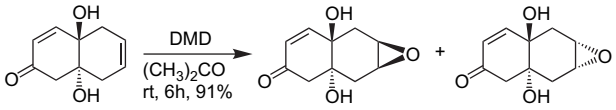
into a variety of stereospecifically oxygenated compounds. One of the common reactions of unsaturated bicyclic endoperoxides is the thermal cleavage of the weak oxygen–oxygen bond followed by addition of the oxygen radicals to the adjacent double bond to give diepoxides with a *syn*-configuration. Unsaturated bicyclic endoperoxides can be conveniently converted into the corresponding diepoxides by using cobalt(II) tetraphenylporphyrin (Co–TPP).[5b,11] Treatment of compound **8** with Co–TPP gave bis-epoxy-alcohol as the sole product **14**, which has a very interesting quercitol skeleton (Scheme 6).



Scheme 6.

The structure of **14** was determined by the spectroscopic data. Olefinic (5.70–5.57 ppm), three epoxide (3.37–2.90 ppm), three methylenic (2.21–1.59 ppm), and alcoholic protons (2.40 ppm) were seen in the ^{1}H NMR spectrum. The ^{13}C NMR spectrum showed two signals (127.1, 125.3 ppm) in the olefinic area and eight signals (71.5, 59.9, 55.1, 50.4, 47.5, 40.2, 36.7, 32.0 ppm) in the saturated hydrocarbon area. The COSY experiment is also fully consistent with this structure (**14**).

For further chemical transformation, we decided to convert **9** to its corresponding epoxide. Mono-epoxidation of **9** with dimethyloxirane[12] gave an isomeric mixture of **15a**, **15b** (Scheme 7) as expected. Unfortunately, the attempt of separation of this mixture by chromatographic and crystallization technique was unsuccessful. The structures of **15a** and **15b** were elucidated by ^{1}H and ^{13}C NMR spectrum. The ^{1}H NMR spectrum showed two signals at 6.70 and 6.62 ppm belonging to β-protons of α,β-unsaturated double bonds. Olefinic protons (6.70–5.98 ppm), epoxide protons (3.55–3.40 ppm), three methylenic protons (2.96–2.90, 2.52–2.28, 2.14–2.09 ppm), and alcoholic protons (5.00 ppm) were seen in the ^{1}H NMR spectrum as a mixture of isomers **15a** and **15b**.



Scheme 7.

Especially, the ^{13}C NMR spectrum showed four signals (149.6, 148.8, 128.2, 127.9 ppm) in the olefinic area. Two carbonyl carbons resonated at 199.3 and 199.2 ppm. As seen, it is not possible to reach the conclusion about which signals belonged to which isomers.

3. Conclusion

In conclusion, we synthesized (1*R**,6*R**,8*R**)-9,10-dioxatricyclo[6.2.2.0^{1,6}]dodeca-3,11-dien-6-yl hydroperoxide **7** and investigated for the first time its further chemical transformation with dimethylsulfide, NEt_3, PPh_3, and Co–TPP, respectively. We believe that these finding can be used as key compounds for the synthesis of oxyfunctional decalin derivatives.

4. Experimental section

4.1. General

Column chromatography: *Silica gel 60* (70–230 mesh) and *neutral Al$_2$O$_3$* (III activated). Solvents was purified and dried by standard procedures before use. Melting point: *Büchi-539* cap. melting point apparatus; uncorrected. IR Spectrum (KBr): *Mattson-1000 FT-IR* spectrophotometer; in cm^{-1}. ^{1}H and ^{13}C NMR spectrum: *Varian* spectrometers. Elemental analyses were carried out with a *Leco CHNS-932* instrument. High resolution mass spectra for all the new compounds were done by micrOTOF (*Bruker*).

4.1.1. Reaction of 1,4,5,8-tetrahydronaphthalene **5** with singlet oxygen (**7**)

To a stirred solution of 1,4,5,8-tetrahydronaphthalene (**5**) (2.0 g, 15.15 mmol) in 150 mL of CH_2Cl_2 was added 20 mg of tetraphenylporphyrin (TPP). The resulting mixture was irradiated with a tungsten halogen projection lamp (500 W) while oxygen was being passed through the solution and the mixture was stirred at room temperature for 2 h. Evaporation of the solvent in vacuo (80% yield according to ^{1}H NMR), and chromatography of the residue on Al_2O_3 column (20 g, jacket column) (20% ethyl acetate/hexanes) gave pure 9,10-dioxatricyclo[6.2.2.0^{1,6}]dodeca-3,11-dien-6-yl hydroperoxide (**7**). It was crystallized from CH_2Cl_2/hexanes (2.0 g, 68%) as a colorless solid, mp: 103–104 °C. [Found: C, 55.71; H, 7.08. $C_{10}H_{12}O_4$ requires: C, 55.81; H, 7.02%]; δ_H (400 MHz, $CDCl_3$, ppm) 7.64 (1H, s, OO*H*), 6.72 (1H, dd, *J* 8.4, 6.2 Hz, C*H*=), 6.25 (1H, dd, *J* 8.4, 1.5 Hz, C*H*=), 5.68–5.61 (2H, m, C*H*=C*H*), 4.76 (1H, m, C*H*O–O), 2.88 (1H, dd, *J* 18.8, 5.1 Hz, CH=CH–CH_2), 2.48 (1H, m, CH=CH–CH_2), 2.59–2.36 (2H, m, CH=CH–CH_2), 2.14 (1H, dd, *J* 13.5, 4.0 Hz, CH_2–CHO–O), 2.02 (1H, dd, *J* 13.9, 1.6 Hz, CH_2–CHO–O). δ_C (100 MHz, $CDCl_3$, ppm) 134.2, 132.8, 124.5, 122.8, 79.2, 75.1, 72.0, 37.2, 32.6, 30.1. IR ($CHCl_3$, cm^{-1}): 3400, 3033, 2936, 2902, 1662, 1417, 1374, 1237, 1092, 851.

4.1.2. Reduction of **7** with Me_2S (**8**)

To magnetically stirred slurry of 410 mg (5.4 mmol) of Me_2S in 25 mL of CH_2Cl_2 was added a solution of 500 mg (2.55 mmol) of endoperoxide **7** in 25 mL of CH_2Cl_2 at room temperature. After the addition was complete (ca. 10 min), the mixture was stirred for 6 h, CH_2Cl_2 was evaporated in vacuo, the residue was extracted with CH_2Cl_2 (3×30 ml), and the extract was dried (Na_2SO_4). The solution was evaporated in vacuo and gave pure 9,10-dioxatricyclo[6.2.2.0^{1,6}]dodeca-3,11-dien-6-ol **8** (322 mg, 70%, solid). It was crystallized from dichloromethane/hexane as a pale yellow solid, mp: 64–65 °C. δ_H (400 MHz, $CDCl_3$, ppm) 6.78–6.74 (1H, dd, *J* 8.4, 4.4 Hz, CH=C*H*), 6.31 (1H, d, *J* 8.4, 1.8 Hz, C*H*=CH), 5.67 (2H, m, CH$_2$–C*H*=C*H*), 4.68 (1H, m, C*H*–O–O), 2.7–2.65 (2H, m, CH_2–CH=CH–CH_2), 2.43–2.34 (2H, m, CH_2–CH=CH–CH_2), 2.28–2.24 (1H, dd, *J* 13.6, 4.0 Hz, C*H*–CHO), 1.66–1.63 (1H, d, *J* 13.6 Hz, C*H*–CHO). δ_C (100 MHz, $CDCl_3$, ppm) 134.7, 133.8, 124.7, 123.4, 76.8, 72.1, 66.8, 41.8, 38.0, 29.7. IR ($CHCl_3$, cm^{-1}): 3498, 3032, 2965, 2902, 2819. HRMS: (ESI/[M+Na]$^+$) *m*/*z* found: 203.1113, requires: 203.0679.

4.1.3. 4α,8α-Dihydroxy-4α,5,8,8α-tetrahydro-1H-naphthalen-2-one (**9**)

To a magnetically stirred solution of mono alcohol **8** (1 g, 5.56 mmol) in 50 mL of CH_2Cl_2 at 0 °C was added NEt_3 (0.58 g, 5.75 mmol, 0.8 mL). The reaction mixture was stirred at room temperature for 5 h. The solvent was removed under reduced pressure and the residue was purified by column chromatography (35 g neutral alumina), eluting first with CH_2Cl_2 and then with ethyl acetate. The ethyl acetate fraction afforded pure 4α,8α-dihydroxy-4α,5,8,8α-tetrahydro-1*H*-naphthalen-2-one (**9**) (0.7 g, 70%). Colorless solid from ethyl acetate/dichloromethane, mp: 123–124 °C. [Found: C, 67.08; H, 6.76. $C_{10}H_{12}O_3$, requires: C, 66.65; H, 6.71%]; δ_H (400 MHz, CD_3OD, ppm): 6.66 (1H, d, *J* 9.9 Hz, O=C–C*H*=CH), 5.94 (1H, d, *J* 9.9 Hz, O=C–CH=C*H*), 5.63 (2H, m, CH$_2$–C*H*=C*H*–CH$_2$), 4.85 (2H, br s, O*H*), 2.91 (1H, d, *J* 16.5 Hz, O=C–CH_2), 2.51 (2H, m, CH_2–CH=CH–CH_2), 2.16 (1H, d, *J* 16.9 Hz, CH_2–CH=CH), 1.99 (1H, d, *J* 16.9 Hz, CH=CH–CH_2), 2.36 (1H, d, *J* 16.5 Hz, O=C–CH_2). δ_C

(100 MHz, CD_3OD, ppm): 200.0, 150.1, 128.2, 123.6, 123.4, 72.6, 68.0, 46.6, 35.4, 34.1. HRMS: (M^+) *m*/*z* found: 181.0865, requires: 181.0820.

4.1.4. Co–TPP reaction of ***8***

To a magnetically stirred solution of (3 mmol, 540 mg) endoperoxide **8** in 30 mL of CH_2Cl_2 was added a solution of 20 mg (0.04 mmol) of cobalt *meso*-tetraphenylporphyrin[13] in 5 mL of CH_2Cl_2 at 0 °C. After the addition was completed (15 min), the mixture was stirred for 1 h at room temperature. The solvent was rotoevaporated (15 mmHg, 25 °C). The residue was chromatographed on Al_2O_3 eluting first with dichloromethane/*n*-hexane (1:3) and then with pure CH_2Cl_2. The CH_2Cl_2 fraction afforded pure **14** (486 mg, 90%). Colorless solid from ethyl acetate/dichloromethane, mp: 80–81 °C. [Found C, 66.50; H, 6.56. $C_{10}H_{12}O_3$ requires: C, 66.65; H, 6.71%]; δ_H (400 MHz, $CDCl_3$, ppm): 5.7–5.57 (2H, m, C*H*═C*H*), 3.37 (m, 1H, C*H*O–CO–), 3.28 (m, 1H, C*H*O–CHO), 2.99 (m, 1H, CHO–C*H*O), 2.89 (dm, 1H, *J* 19.9 Hz, –C*H*$_2$–), 2.40 (s, 1H, OH), 2.20 (m, 2H, –C*H*$_2$–), 2.04 (dd, 1H, *J* 15.8, 6.8 Hz, –C*H*$_2$–), 1.77 (d, 1H, –C*H*$_2$–, *J* 19.9 Hz), 1.59 (dd, 1H, *J* 15.8, 2.6 Hz, –C*H*$_2$–). δ_C (100 MHz, $CDCl_3$, ppm): 125.1, 123.5, 69.5, 58.1, 53.2, 48.7, 45.8, 38.3, 35.0, 30.2. IR ($CHCl_3$, cm^{-1}): 3466, 3029, 2927, 2895. HRMS: (ESI/$[M+Na]^+$) *m*/*z* found: 203.1155, requires: 203.0684.

4.1.5. Deoxygenation of the hydroxy-epoxide ***8*** *with triphenylphosphine (****11****)*

To a magnetically stirred solution of the 9,10-dioxatricyclo[6.2.2.01,6]dodeca-3,11-dien-6-ol **8** (220 mg, 0.99 mmol) in 25 mL of CH_2Cl_2 at 0 °C was added dropwise a solution of triphenylphosphine (260 mg, 0.99 mmol) in 10 mL of CH_2Cl_2 over 1 h. After the addition was completed, the reaction mixture was stirred for additional 1 h at room temperature. Evaporation of the solvent (30 °C, 20 mmHg) and chromatography of residue on a neutral alumina (20 g, Al_2O_3, activity-III), eluting with 25% ethyl acetate/hexane gave the 1a,4,5,8-tetrahydro-1-oxa-cyclopropa[*d*] naphthalene-4a-ol (**11**) (115 mg, 60%, yellow liquid). δ_H (200 MHz, $CDCl_3$, ppm): 6.00 (1H, ddt, *J* 9.7, 3.9, 1.9 Hz, C*H*═CH), 5.85 (1H, ddd, *J* 9.7, 4.0, 1.7 Hz, –CH═C*H*–), 5.77–5.64 (2H, m, –C*H*═C*H*–), 3.85 (1H, dd, *J* 3.9, 1.7 Hz, C*H*–O), 3.06 (1H, dm, *J* 19.1 Hz, C*H*$_2$–CH═CH), 2.37–2.21 (4H, m, C*H*$_2$–CH═CH–C*H*$_2$), 2.09 (1H, m, O*H*), 1.81 (1H, dm, *J* 19.1 Hz, C*H*$_2$–CH═CH–CH–O); δ_C (50 MHz, $CDCl_3$, ppm): 132.3, 127.2, 125.8, 125.7, 69.9, 63.3, 55.7, 40.2, 39.0, 32.3. IR (KBr, cm^{-1}): 3460, 3032, 2977, 2907, 2819. HRMS: (ESI/$[M+Na]^+$) *m*/*z* found: 186.4978, requires: 187.0730.

4.1.6. Dimethyldioxirane epoxidation of ketone ***9***

To a magnetically stirred solution of 100 mL, 0.07 M of dimethyldioxirane (DMD) in acetone was added 200 mg (0.260 mmol) of ketone **9** and the resulting mixture was stirred at room temperature (ca. 20 °C) for 24 h, while the reaction progress was monitored by means of the peroxide test (KI/HOAc). After evaporation of the solvent the mixture was chromatographed on silica gel (30 g) with 2% methanol/ethyl acetate as eluent to afford 20 mg unreacted ketone **9** and 198 mg (91%) epoxide mixture **15** as yellow liquid. [Found: C, 61.78; H, 6.55. Required C, 61.22; H, 6.16%]; HRMS: (M+) *m*/*z* found: 197.0812, requires: 197.0786.

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