Synthesis based on cyclohexadienes. Part 24. A new total synthesis of pupukean-2-one and a facile entry to copa and ylanga type sesquiterpene skeletons

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A novel tandem 5-exo-trig allyl and 3-exo-trig radical cyclisation and rearrangement to copa and ylanga type sesquiterpene skeletons from easily prepared cyclohexadienes is reported. A new total synthesis of pupukean-2-one, which belongs to a novel class of sesquiterpenes, involving a 5-exo-trig allyl radical cyclisation as the key step is also reported.

In the total synthesis of 5-epi-pupukean-2-one described in the preceding paper, the diene ester was utilised as an equivalent of the substituted 1-methylcyclohexa-1,3-diene. Substituted 1-methoxycyclohexa-1,3-dienes, readily prepared by the metal-ammonia reduction of the corresponding aromatic ethers followed by a base-catalysed conjugation, afford regio-specific adducts of the type upon cycloaddition with a ketene equivalent. If the bicyclic ketone can be transformed into the bicyclic ketone, then the dihydro compound can be used as an equivalent of 1-methoxycyclohexa-1,3-diene which essentially involves bridgehead substitution of the methoxy group by a methyl group.

Although this bridgehead substitution methodology has been reported earlier, its application to the synthesis of natural products has been dismal. We have investigated this reaction which involved the tricyclic compounds having a bridgehead methoxy group that led to the total synthesis of (±)-cedrol (khusiol). In continuation of our interest in the total synthesis of sesquiterpenes, involving the bridgehead substitution strategy, we describe herein a new total synthesis of pupukean-2-one through a 5-exo-trig allyl radical cyclisation and a novel tandem 5-exo-trig allyl and 3-exo-trig radical cyclisation and its rearrangement to copa and ylanga type sesquiterpene skeleton.

Results and discussion

The total synthesis of pupukean-2-one was devised using the bridgehead substitution strategy as depicted in Scheme 1.

The retrosynthetic analysis indicated that pupukean-2-one can be obtained from the tricyclic ketone which, in turn, can be prepared from the enone through a bridgehead substitution strategy. The tricyclic ketone can be obtained from the bicyclic ketone having the prenyl group in the endo position which, in turn, can be made from the known bicyclic ketone.

Synthesis of the bicyclic ketone

Thus, the bicyclic ketone upon alkylation with LDA and Mel afforded the ketone having the methyl group in the endo position (Scheme 2), as evidenced by the spectral data. The "H NMR spectrum of showed a doublet at δ 1.09 for the endo methyl group, a multiplet at δ 2.73 for the bridgehead proton.

and a singlet at δ 3.52 for the bridgehead methoxy group. The 
$^{13}$C NMR spectrum of 13 showed only 10 lines which supports 
the stereoselective nature of the alkylation. Further alkylation 
with prenyl bromide was achieved by treating the ketone 13 
with LDA at $-78^\circ$C and quenching the resultant enolate with 
prenyl bromide in HMPA–THF. The $^1$H NMR spectrum of the 
resulting ketone 11 showed signals at δ 1.08 (s, Me), 1.59 and 
1.73 (both s, allylic Me) and 5.12 (prenyl olefinic H).

The bicyclic ketone 11 having been prepared, the next step 
was its acid-catalysed rearrangement to generate the tricyclic 
system. This was achievable through an intramolecular ene 
cyclisation of the unsaturated ketone 14, obtained from 11, to 
afford the tricyclic enone 15 (Scheme 3). The resulting tricyclic 
enone 15 was convertible into pupukean-2-one 8.

With this idea, we attempted a novel one-pot tandem acid-
catalysed rearrangement and an intramolecular ene cyclisation 
of the bicyclic ketone 11: all attempts failed. Thus, treatment of 
the bicyclic ketone 11 with Lewis acid BF$_3$·MeOH, at room 
temperature gave only recovery of starting material, whilst for 
reactions at higher temperatures, the product decomposed. 
Treatment of 11 with SnCl$_2$, BF$_3$·OEt$_2$ and HCO$_2$H failed to 
produce the desired compound as did heating it with PTSA in 
refluxing benzene.

Stereo- and regio-selective alkylation
Since the acid-catalysed cyclisation of 11 failed to yield the 
tricyclic skeleton, a radical cyclisation$^9$ was investigated for the 
key step to the tricyclic skeleton 15; this was a new strategy 
involving a 5-exo-trig allyl radical cyclisation.$^{10}$

In this approach, the allyl bromide 18 was considered as the 
key intermediate (Scheme 4), since it can undergo an intra-
molecular 5-exo-trig allyl radical cyclisation to give the iso-
twistane 17 which can be elaborated to pupukean-2-one 8.

Although allyl radicals have been known for a decade,$^{10}$ their 
cyclisation has been rarely used synthetically,$^{11,12}$ since their 
greater stability makes them less reactive when compared to 
their saturated and vinylic counterparts. With this in mind, allyl 
radical cyclisation of the bromide was investigated as a model 
system.

Since attempted allylic bromination of the ketone 11 with N-
bromosuccinimide afforded a complex mixture of products, 
alkylation of the ketone 13 with the dibromide$^{13}$ 20 was 
investigated.

Alkylation of the lithium enolate of the ketone 13 at $-78^\circ$C 
with 1,4-dibromo-2-methylbut-2-ene$^{14}$ 20, proceeded regio- 
and stereo-selectively to give, exclusively, the endo bromide 19 
(Scheme 5), as evidenced from its spectral data. The $^1$H NMR 
spectrum of 19 showed signals at δ 6.4 (m, olefinic H), 5.73 (t, 
olefinic H) and 3.89 (s, CH$_2$Br); the mass spectrum showed 
peaks at 313 (M$^+$) and 315 (M$^+$ + 2) with base peaks at 
233 and 110.

The alternative structure 21a for the product was ruled out 
on the basis of the NMR data, in particular the coupling of the 
CH$_2$Br protons. Further attempted radical cyclisation (see 
below) of the product gave the ketone 11 as one of the products. 
If 21a was formed during the alkylation of 13 with 20, the 
reduced product would have structure 21b whose NMR 
spectrum would show distinctive signals for the two vinyl methyl 
groups as a singlet and a doublet. The NMR spectrum of the 
product was consistent with structure 11 and hence the regio-
selectivity in the alkylation using the dibromide 20. The reasons 
for this regioselectivity is not very clear but appears to be due to 
the steric hindrance from the methyl group which is being exam-
ined now.

A novel radical rearrangement
The allyl bromide 19 having been successfully obtained in good 
yield, its intramolecular allyl radical cyclisation was inves-
tigated (Scheme 6). Radical cyclisation under standard con-
ditions$^{16}$ (0.005 M benzene solution of 19 with 1.1 equiv. of 
TBTH and 0.1 equiv. of AIBN, reflux, 1–2 h) afforded a mixture 
containing the reduced product 11 (5%) and a new com-
 pound 22 (60%) whose IR spectrum showed absorption at 1740 
cm$^{-1}$; in its $^1$H NMR spectrum signals for olefinic protons at 
δ 6.2, 6.4 and 5.1 were absent, but instead there were signals at 
δ 4.6 and 4.8 (both d). This clearly suggested that an intra-
molecular cyclisation has occurred. Since $^{13}$C NMR spectrum of 
22 showed a methine carbon at δ 78.2 the compound is dif-
f erent from the expected 5-exo-trig allyl radical cyclisation 
product 23. On treatment with PTSA, compound 22 was quan-
titatively converted into a new isomer 24, whose IR spectrum 
showed carbonyl absorption at 1740 cm$^{-1}$; moreover, its $^1$H 
NMR spectrum showed no signals for olefinic protons, suggest-
ing that the \textit{exo} olefin is isomerised into a stable tetrasubstituted olefin. The off-resonance $^{13}$C NMR spectrum of 24 showed the presence of four singlets, four doublets, three triplets and four quartets, whereas the expected product 10 should have five singlets, two doublets, four triplets and four quartets. Also, a doublet at $\delta$ 78.53 clearly showed that OMe is attached to a carbon atom bearing a hydrogen.

These data clearly established the structure of the cyclised and isomerised products as 22 and 24 respectively (Scheme 6) and that the isopropenyl substituent present in 22 was isomerised to the isopropylidene group under acidic conditions to give 24. A probable mechanism for the formation of these compounds 22 and 24 is indicated (Scheme 7). As expected, the initial 5-\textit{exo-trig} allyl radical cyclisation gave the radical 26 which underwent a 3-\textit{exo-trig} radical cyclisation onto the carbonyl group to give the cyclopropyloxyl radical 27 which further rearranged to 22. Formation of a stable radical adjacent to the methoxy group appeared to be the driving force for this arrangement.

The proposed mechanism was further confirmed by oxidative cleavage of 24 to the diketone 29 (Scheme 8) whose IR spectrum showed absorption at 1740 cm$^{-1}$ (five-membered ring with a keto group) and in whose $^1$H NMR spectrum olefinic protons signals were absent. Hydrogenation of 22 afforded a saturated tricyclic ketone 30 in whose $^1$H NMR spectrum olefinic protons signals were absent.

A number of natural products possess this skeleton, e.g. copacamphor 31,14 sinulurene 3215 and sativene 3316 (Scheme 9) and their total synthesis by the above strategy may be envisaged. Interestingly, pfaffic acid 34,17 a nortriterpene with many biologically interesting properties was found to possess this skeleton as part of the DEF ring system.

**Total synthesis of pupukean-2-one 8**

Since the intramolecular 5-\textit{exo-trig} allyl radical cyclisation of 19 resulted in the tricyclic compound 22, presumably through the isotwistane intermediate 27, because of the stabilisation of the radical at the carbon bearing the methoxy group, it was expected that the radical cyclisation of 18 should result in the desired isotwistane moiety 17.

Thus, the ketone 12 on treatment with PTSA in refluxing benzene, afforded the enone 35, whose structure was deduced from the spectral data. The enone 35 upon treatment with MeLi...
in ether afforded the tertiary alcohol 36, whose IR spectrum showed the disappearance of carbonyl group absorption and the appearance at 3300 cm\(^{-1}\) of hydroxy group absorption. The tertiary alcohol 36 upon treatment with a catalytic amount of perchloric acid afforded the bicyclic ketone 37, whose structure was deduced from the spectral data and was comparable to that reported.\(^{18}\) The IR spectrum of 37 showed strong absorption at 1720 cm\(^{-1}\) (saturated ketone) whilst its \(^1^H\) NMR spectrum showed signals at \(\delta\) 1.22 (s, bridgehead Me) and 2.94 (bridgehead H) but no methoxy group signal at \(\delta\) 3.5. The \(^{13}C\) NMR spectrum of 37 showed a quartet, three triplets, three doublets and two singlets confirming the above structure. The mass spectrum showed its base peak at 94 due to the diene formed by the loss of a ketene due to retro Diels–Alder fragmentation. Alkylation of the lithium enolate generated from the bicyclic ketone 37, with methyl iodide afforded exclusively the ketone 38 having an \textit{endo} methyl group (Scheme 10); its \(^1^H\) NMR spectrum showed only one doublet at \(\delta\) 1.02 (\textit{endo}-Me) whilst its mass spectrum showed a base peak at 94 due to the retro Diels–Alder fragment and a molecular ion peak at 150. Further alkylation of the ketone 38 with 1,4-dibromo-2-methylbut-2-ene 21 gave the bicyclic allyl bromide 18 having a five-carbon substituent in the \textit{endo} position; its \(^1^H\) NMR spectrum showed signals at \(\delta\) 5.8 (prenyl olefinic H), 3.98 (s, CH\(_2\)Br) and 1.73, 1.21 and 1.05 (all s, Me). The mass spectrum of 18 showed a very weak molecular ion peak at 297 (M\(^+\)), a strong peak at 217 (M\(^+\)) and a base peak at 94 as the peak formed by the loss of a ketone due to retro Diels–Alder fragmentation. Alkylation of the lithium enolate generated from the bicyclic ketone 37, with methyl iodide afforded exclusively the ketone 38 having an \textit{endo} methyl group (Scheme 10); its \(^1^H\) NMR spectrum showed only one doublet at \(\delta\) 1.02 (\textit{endo}-Me) whilst its mass spectrum showed a base peak at 94 due to the retro Diels–Alder fragment and a molecular ion peak at 150. Further alkylation of the ketone 38 with 1,4-dibromo-2-methylbut-2-ene 21 gave the bicyclic allyl bromide 18 having a five-carbon substituent in the \textit{endo} position; its \(^1^H\) NMR spectrum showed signals at \(\delta\) 5.8 (prenyl olefinic H), 3.98 (s, CH\(_2\)Br) and 1.73, 1.21 and 1.05 (all s, Me). The mass spectrum of 18 showed a very weak molecular ion peak at 297 (M\(^+\)), a strong peak at 217 (M\(^+\)) and a base peak at 94 as the peak formed by the loss of a ketone due to retro Diels–Alder fragmentation.

In conclusion, an efficient method for the construction of the copa and ylanga type sesquiterpene skeleton is reported. This methodology is fairly flexible and can be extended for the total synthesis of pfafic acid, a highly biologically active norrterpene. A new total synthesis of pupukean-2-one 8, which belongs to a novel class of sesquiterpenes, involving a 5-\textit{exo-trig}-allyl radical cyclisation as the key step is also reported.

\section*{Experimental}

\subsection*{1-Methoxy-3-\textit{endo}-methylbicyclo[2.2.2]oct-5-en-2-one 13}

A 1 mol dm\(^{-3}\) solution of BuLi in hexane (21.7 cm\(^3\), 21.7 mmol) was added to diisopropylamine (2.9 cm\(^3\), 22 mmol) in THF (30 cm\(^3\)) at \(-78^\circ\text{C}\) under argon. The resultant solution of lithium diisopropylamide was stirred at \(-78^\circ\text{C}\) for 1 h after which a solution of the ketone 12 (3 g, 19.7 mmol) in dry THF (40 cm\(^3\)) was added dropwise to it. The resultant lithium enolate was stirred at \(-78^\circ\text{C}\) for 1 h after which a solution of MeI (2.5 cm\(^3\), 40 mmol) in THF was added at once. After being stirred for 1 h, the reaction mixture was poured onto saturated aqueous \(\text{NH}_4\text{Cl}\) and extracted with ether (3 \(\times\) 50 cm\(^3\)). The combined extracts were washed successively with water, \(\text{aq. sodium thiosulfate, water and brine, dried (Na}_2\text{SO}_4}\) and evaporated. Purification of the residue using column chromatography on [ethyl acetate-light petroleum (1:9)] afforded the product 13 as a colourless liquid (2.9 g, 90\%); \(v_{\text{max}}\)\text{cm}^{-1} 2930, 1715 and 1640; \(\delta_n\) 1.09 (3H, d, J 7.2, CH\(_3\)Me), 1.6–2.2 (5H, m), 2.73 (1H, m, bridgehead H), 3.52 (3H, s, OMe) and 6.1–6.5 (2H, m, olefinic H); \(\delta_c\) 16.7 (q), 24.4 (t), 24.98 (t), 37.56 (d), 43.41 (d), 52.26 (q), 83.6 (s), 129 (d), 134.19 (d) and 210.34 (s); \(m/z\) 166 (M\(^+\), 55\%), 138 (100), 122 (100) and 110 (100) (Found: M\(^+\), 166.0986. \(C_{10}H_{12}O_2\) requires \(M\) 166.0994).

\subsection*{1-Methoxy-3-methyl-3-\textit{endo}-(3-methyl-2-but-2-ene)bicyclo-\[2.2.2\]oct-5-en-2-one 11}

To a freshly prepared LDA solution [prepared from a 1 M solution of BuLi (8.6 cm\(^3\), 8.6 mmol) and diisopropylamine (1.2 cm\(^3\), 9.4 mmol) in THF (20 cm\(^3\))] at \(-78^\circ\text{C}\) under argon, was added dropwise a solution of the ketone 13 (1.3 g, 7.8 mmol) in THF (30 cm\(^3\)). The resultant solution was stirred for 1 h at the same temperature after which it was quenched with a solution of prenyl bromide (1.7 cm\(^3\), 15 mmol) in THF and then treated with HMPA (2.8 cm\(^3\), 15 mmol). The reaction mixture was stirred overnight and then poured onto 2 M aq. HCl (100 cm\(^3\)). Work-up followed by column chromatography on [ethyl acetate-light petroleum (1:9)] afforded compound 11 as a colourless oil (1.35 g, 80\%); \(v_{\text{max}}\)\text{cm}^{-1} 3020, 2940, 1720 and 1640; \(\delta_n\) 0.85–2.18 (6H, m), 1.08 (3H, s, Me), 1.59 (3H, s, Me), 1.73 (3H, s, Me), 2.61 (1H, m, bridgehead H), 3.52 (3H, s, OMe), 5.12 (1H, t, J 7.1 Hz, olefinic H), 6.17 (1H, dd, J 6.7 and 1.7, olefinic H) and 6.45 (1H, dd, J 8.2 and 6.7); \(\delta_c\) 17.6 (q), 21.0 (q), 21.1 (q), 25.7 (t), 26.2 (t), 36.5
1,4-Dibromo-2-methylbut-2-ene 20
To a stirred solution of triphenylphosphane (0.25 mmol) in anhydrous toluene (10 ml) at room temperature, was added dropwise bromine (0.5 ml). The resulting solution was stirred for 1 h at the same temperature and then quenched with a solution of sodium thiosulfate (3 g) in water (20 ml). The reaction mixture was then extracted with ether (3 x 25 ml) and the combined extracts were dried (Na2SO4) and evaporated. The residual oil was distilled to give a colourless crystalline solid; mp 92°C; v_max/cm^-1 2920, 2930, 1740 and 1720; δ_H(3H, Me), 1.85 (3H, s, Me), 3.95 (2H, d, J 7.2, H_HCCMe) and 5.92 (1H, t, J 8.1, olefinic H).

5-Methoxy-8-methyltricyclo[6.2.1.0^12]undeca-7-one 22
A solution of the ketone 12 (3 g, 23 mmol) and PTSA (4 g) in dry benzene (10 ml) was refluxed under nitrogen atmosphere for 2 h after which it was diluted with benzene and washed with water, saturated aq. sodium hydroxide and then dried (Na2SO4). Chromatography of the residue on silica gel using ethyl acetate-hexane (1:1) eluent gave the tricyclic olefin 12 as a colourless oil (53 mg, 60%); v_max/cm^-1 2920 and 1742; δ_H(3H, Me), 1.1–2.2 (9H, m), 3.38 (3H, s, OMe) and 3.46 (1H, m, CHOMe); m/z 208 (M^+ 28%), 148 (35) and 128 (100) (Found: M^+, 208.1097. C_{12}H_{12}O_4 requires M^+, 208.1099).

1-Methoxybicyclo[3.2.1]oct-3-en-2-one 35
A solution of the ketone 12 (3 g, 23 mmol) and PPTS (4 g) in dry benzene was refluxed under nitrogen atmosphere for 2 h. Excess of methyl-lithium was quenched by addition of saturated aq. ammonium chloride. Work-up afforded the alcohol 36 as a colourless liquid (2.2 g, 83%); v_max/cm^-1 3030–2930; δ_H(3H, Me), 1.1–2.5 (9H, m), 1.3 (3H, s, Me), 3.35 (3H, s, OMe) and 5.29 (1H, d, J 9.4, olefinic H), 5.6 (1H, dd, J 9.4 and 5.2, olefinic H).

1-Methylbicyclo[2.2.2]oct-5-en-2-one 37
A mixture of the alcohol 36 (12 g, 11.9 mmol), CH_2Cl_2 (50 cm^3) and HClO_4 (70% aqueous; 3 drops) was stirred at room temperature. After being stirred for 1 h, the reaction mixture was evaporated in vacuo and the residue was distilled under reduced pressure (bp 60°C/10 mmHg) to afford the dibromide 20 as a colourless oil (10 g). δ_H(3H, Me), 1.85 (3H, s, Me), 3.93 [2H, s, H_HCCMe]; 3.95 (2H, d, d, J 7.2, H_HCCMe) and 5.92 (1H, t, J 8.1, olefinic H).

3-endo-4-Bromo-3-methyl-2-enyl-1-methoxy-3-methylbicyclo[2.2.2]oct-5-en-2-one 19
To a freshly prepared LDA solution (prepared from 1 mmol of LDA in THF and 1 mmol of dry ether) at room temperature, was added dropwise a solution of the tricyclic ketone 13 (1 g, 3.19 mmol) in THF (20 ml). The resultant solution was stirred for 1 h at the same temperature and then quenched with a solution of 1,4-dibromo-2-methylbut-2-ene 21 (1.5 g, 0.51 mmol) in THF and then treated with HMPA (1.2 ml, 6.5 mmol). The reaction mixture was then stirred overnight and poured onto 2 ml of aq. HCl (100%). Work-up followed by column chromatography [ethyl acetate-light petroleum (1:9)] afforded the bromo ketone 19 as a colourless oil (1.3 g, 70%); v_max/cm^-1 3010, 2920, 1720 and 1640; δ_H(3H, s, Me), 1.2–2.36 (6H, m), 1.76 (3H, s, Me), 2.62 (1H, m, bridgehead H), 3.52 (3H, s, OMe), 3.99 (2H, s, CH_2Br), 5.73 (1H, t, J 6.8, olefinic H), 6.21 (1H, dd, J 6.4 and 1.8, olefinic H) and 6.51 (1H, dd, J 8.1 and 6.4, olefinic H); δ_C 14.6, 21.7, 21.8, 26.4, 36.8, 39.9, 40.8, 46.9, 52.7, 84.1, 125.5, 127.6, 134.4 and 126.4; m/z 315 (M^+ + 5%, 313 (M^+), 233 (30), 205, 170, 137 (100) and 110 (100) (Found: M^+ – Br, 233.1523. C_{10}H_{12}O_4 requires M^+, 233.1540).
perature for 30 min after which it was diluted with CH₂Cl₂, washed with aqueous sodium carbonate and brine and then dried (Na₂SO₄) and evaporated. Chromatography of the crude product over silica gel [ether–pentane (1:49) as eluent] yielded the ketone 37 as a viscous liquid (1.3 g, 82%); ν max/cm⁻¹: 3010, 2925, 1720 and 1630; δ₁H 1.22 (3H, s), 1.2–2.22 (4H, m), 2.05 (2H, d, J, CH₂CO), 2.95 (1H, m, bridgehead H), 5.67 (1H, d, J 8.2 and 1.6, olefinic H) and 6.47 (1H, dd, J 8.2 and 6.6, olefinic H); δ₁C 17.3 (q), 25.6 (t), 29.8 (t), 31.8 (d), 40.0 (t), 48.5 (s), 133.3 (d), 136.6 (d) and 212.6 (s); m/z: 136 (M⁺), 10% and 94 (100) (Found: M⁺, 136.0890. CH₃H₂O requires M⁺, 136.0888).

1,3-Dimethylbicyclo[2.2.2]oct-5-en-2-one 38

To a freshly prepared LDA solution [prepared from 1 mol dm⁻³ solution of BuLi (7.2 cm³, 7.2 mmol) and diisopropylamine (1 cm³, 7.9 mmol) in THF (20 cm³)] at −78 °C under argon, was added dropwise a solution of the ketone 37 (19, 6.57 mmol) in THF (20 cm³). After being stirred at −78 °C for 1 h, the reaction mixture was refluxed for 3 h, the reaction mixture was concentrated under reduced pressure. The residue was purified over a silica gel column [ethyl acetate–hexane (1:19)] to furnish the tricyclic olefin 9 (70 mg, 90%) which was recrystallised from hexane; ν max/cm⁻¹: 2920 and 1712; δ₁H 0.89 (3H, s, Me), 1.17 (3H, s, Me), 0.9–2.4 (9H, m), 1.52 (3H, s, Me), 1.62 (3H, s, Me) and 2.9 (1H, m, 6-H); m/z: 218 (M⁺, 100%), 162 (50) and 134 (80) (Found: M⁺, 218.1681. CH₃H₂O requires M⁺, 218.1671).

Pupukean-2-one 8

A suspension of the tricyclic olefin 9 (60 mg, 0.33 mmol) and 10% Pt–C (20 mg) in dry MeOH (5 cm³) was magnetically stirred under a H₂ atmosphere for 12 h after which it was filtered through a Celite pad and once again stirred for 2 h under hydrogen after addition of a further catalytic amount of Pt–C. After this, the reaction mixture was filtered through a pad of Celite and evaporated under reduced pressure to furnish the inseparable mixture of the ketone 8 (50 mg) and the isomeric ketone 43 in the ratio (4:1); ν max/cm⁻¹: 1710; δ₁H 0.83 and 0.85 (6H, 2d, 2 × CH₂CH₂H), 0.92 (3H, s, Me), 1.13 (3H, s, Me), 1.3–1.8 (11H, m) and 2.32 (1H, m); m/z: 220 (M⁺, 30%), 159 (80) and 93 (100) (Found: M⁺, 220.1830. Calc. for C₁₅H₂₃O: M⁺, 220.2182).

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References

1. For part 23 of the series; see, K. Kaliappan and G. S. Subba Rao, preceding paper.


3398
20 For a detailed general note, see ref. 1.

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