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BREDT'S RULE—II*

THE SYNTHESIS AND PROPERTIES OF TWO RELATED BRIDGEHEAD ENONES†

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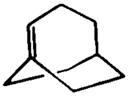
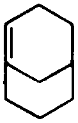
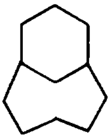
Abstract—Syntheses of bicyclo[5.3.1]undec-7-en-11-one and bicyclo[5.3.1] undec-7(11)-en-8-one are described and the IR, NMR, and UV properties are compared. The chemical properties of the former are also discussed. Evidence is adduced to show that, as predicted, the latter is the more strained and contains a warped double bond.

WISEMAN's restatement‡ of Bredt's rule not only rationalizes, in a simple but satisfying way, the failure of certain reactions which ought to lead to bridgehead olefins, it also allows us to grade such olefins in terms of relative strain. Thus bicyclo[3.2.2]non-1-ene (I), being a bridged *trans*-cycloheptene, ought to be virtually unisolable, whilst the isomer II, as a *trans*-cyclooctene, should be capable of isolation. Experimental evidence bears this out.[1] More importantly, it predicts that although III is isomeric with II it will be enormously strained since it incorporates a *trans*-cyclohexene. Any distinction§ between types II and III was apparently overlooked by Bredt, and Fawcett's S-value formula[2] took no account of it. More recently, significant experimental evidence has emerged from enolization[3] and decarboxylation studies. The object of the work described here, was to prepare and compare isomeric olefins of these types in order to provide a further test of the Wiseman hypothesis. If the Wiseman hypothesis is valid, then the bicyclo[5.3.1]undecane system (IV) should be the smallest which can tolerate bridgehead double bonds in *all three* possible positions. This paper describes the synthesis of the two isomeric αβ-enones VIII and XIX, and an examination of their properties.


I


II


III


IV

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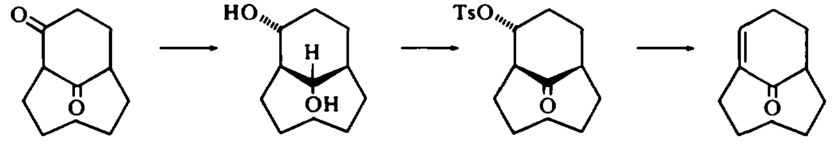
* Part I, Preceding paper.

† This research has been sponsored by the Air Force Office of Scientific Research (O.A.R.) through the European Office of Aerospace Research, United States Air Force.

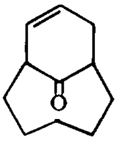
‡ For a synopsis with appropriate references, see Part I.

§ An "intuitive" distinction was made by H. O. House and H. C. Muller, *J. Org. Chem.* 27, 4436 (1962).

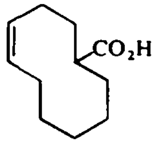
Although a preparation of VIII was described in Part I. it involves a tedious separation of isomers. The pure enone (VIII) is obtained more conveniently by base treatment of the keto-tosylate VII. The preparation of this intermediate from V *via* its hydride reduction product VI has already been described by other workers[5] who assigned it the stereochemistry shown. The effect of base on VII has also been described,[5] but the product was mistakenly formulated as IX. In the light of our experience,[6] it seemed unlikely that an equatorial keto-tosylate of this type would lead to IX. On the other hand, if the tosylate were axial, such a product could be expected, but not under the mild reaction conditions described. A re-examination of the tosylate by Hassner's method[7] showed that it was equatorial (>CH·OTs coupling: $W_{\frac{1}{2}}$ 17 Hz) and on treatment with ethoxide under controlled conditions it afforded the αβ-enone VIII contaminated with 2% of the βγ-isomer IX. Prolonged contact with acid produced the equilibrium mixture of these isomers (75:25 respectively). It is of interest to note that the fragmentation product X was not produced under these conditions although such a process is well known[6a] with smaller ring analogues of VII. The change in reaction path reflects the ability of the bicyclo[5.3.1]undecan-11-one system to sustain a bridgehead carbanion (XI) which leads to VIII by a β-elimination.[*]



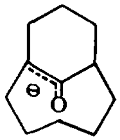
V VI VII VIII



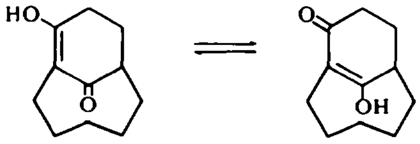
IX



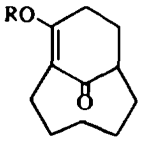
X



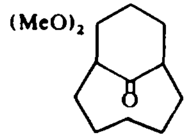
XI



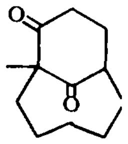
Va



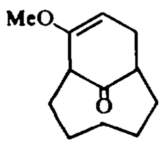
XII R = Ac
XV. R = Me



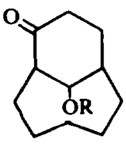
XIII



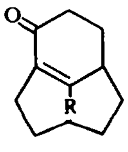
XIV



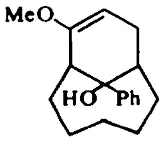
XVI



XVII R = H
XVIII. R = Ms



XIX R = H
XXI. R = Ph



XX

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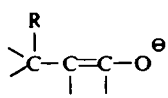
[*] For a similar β-elimination see L. Paquette, *Accounts of Chemical Research* 1, 209 (1968)

The isomeric αβ-enone (XIX) was synthesized from the same β-diketone (V). Other workers have claimed[5] that the spectral properties of V fail to indicate the presence of enol. This is true of the UV spectrum run in hexane, but in EtOH it also shows strong absorption at 268 nm, and on addition of a drop of alkali it absorbs at 315 nm. A knowledge of the composition of equilibrium Va would in itself be interesting, for it would give a (very rough) estimate of the free energy difference* which we are examining. In fact, attempts to trap the enols by acetylation yielded only XII, but this result probably reflects nothing more than the steric congestion around the C(11)—OH.

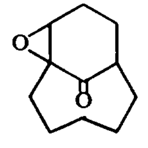
The diketone in MeOH reacted with CH_2N_2 to give a mixture of XV and the C-methyl derivative XIV in the ratio 2:3. The structure of the enol-ether was confirmed by an alternative synthesis (see below) whilst the C-methyl compound showed the NMR (3H: τ 8·84) and doublet ν_{CO} (1720 and 1690 cm^{-1}) features previously described[5] for this compound.† The diketone (V) afforded a monoketal, and in view of the known[5] hindrance at C(11) it was formulated as XIII. This structure was confirmed by its failure to react with either PhMgBr or LAH, and by its pyrolysis to a mixture of methyl ethers (XV and XVI). The latter yielded V on hydrolysis, and showed vinylic absorption in the NMR. The former was identical with the enol-ether obtained above, from CH_2N_2. The mixture of enol-ethers was reduced (LAH) and then hydrolysed to the ketol XVII, whose mesylate (XVIII) yielded the unstable αβ-enone XIX on base treatment. The mixture of enol-ethers also reacted with PhMgBr to give a single product (XX), which was hydrolysed and dehydrated by methanolic acid to the more stable phenyl-enone XXI.

The spectroscopic properties of the isomeric enones VIII and XIX are set out in the Table, together with those of related compounds, for comparison. The IR data are not very informative because ν_{CO} is affected by changes in the C—CO—C bond angle and so they do not lend themselves to ready interpretation. However, the UV spectra are more interesting. It is known that rotation about an essential single bond in a conjugated system leads to a drop in ε_{max} and, for large angles, an accompanying hypsochromic shift. Rotation about an essential double bond produces an intensification of ε_{max} and a bathochromic displacement of λ_{max}.[8] The low ε_{max} of VIII has already been discussed in Part 1 and correlated with the non-planarity of the enone system, but it is significant that λ_{max} is unaffected.‡ Conversely, the near normal ε_{max} and substantial bathochromic shift observed in the *trans*-cyclooctene derivative XIX indicates severe warping of the C=C.§ Unfortunately this substance was not suitable

XXIII



XXIV



XXV

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* An accurate estimate would require a knowledge of the conjugation parameters.

† ν_{CO} splitting is characteristic of most, but not all, non-enolic β-diketones and has been ascribed to dipole interaction. For a review see ref. 6a p. 218.

‡ Even the lower homologue XXII, which must be more distorted, shows[9] an approximately normal λ^{max}.

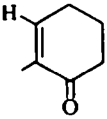
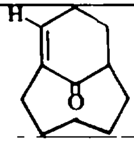
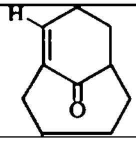
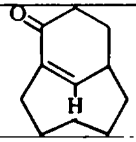
§ For an analogous case, see W. E. Thiessen, H. A. Levy, W. G. Dauben, G. H. Beasley and D. A. Cox, *J. Am. Chem. Soc.* **93**, 4312 (1971).

for X-ray examination, so this fact could not be corroborated: however, it may be taken as experimental proof of Wiseman's claim, that XIX should be more strained than VIII.

The chemical shift of the (β) vinyl proton in these molecules is also informative (see Table). In passing from the planar 2-methylcyclohexenone to VIII and XXII, the *e*-density at the β-carbon rises as the enone becomes less co-planar (i.e. the contribution of XXIIIb to the resonance description of the molecule decreases) and this leads to an upfield shift of the β-H resonance. On the other hand, the marked downfield shift observed in XIX denotes substantial polarization (*cf* XXIIIb) and this is consistent with the hypothesis that double bond character will be energetically "cheaper" at the C(1)—C(10) than at the C(1)—C(11) position.

These conclusions are underscored by the chemical reactivity of the enone isomers. Conjugate addition to VIII should be disfavoured, since it involves an intermediate XXIV, having a strained double bond. We were unable to bring about Michael addition of diethyl malonate to this enone after 48 hr., at room temperature or after a further 70 hr. at 80°. The only new product detected was IX. Epoxidation by means of alkaline perhydrol was slow, and afforded a 1:1 mixture of VIII and XXV after 3 days. The isomer XIX proved to be too unstable for chemical investigation. On brief standing it deposited a colourless solid which is probably a dimer.

TABLE. SPECTROSCOPIC DATA

				
$\nu_C^{CCl_4}$ (cm^{-1})	1670	1702[a] 1690	1710[bc]	1675[f]
λ_{max}^{EtOH} (ε)	235 (10,000)	238 (5630)	238[c] (3000)	254 (7500)
τ (ppm)[d]	3·34	3·84[e]	4·1[c]	3·15

[a] These absorptions are shifted to 1707 and 1697 cm^{-1} in hexane, but since Δ_ν remains constant, they are more probably a C=O doublet than C=O and C=C absorptions, as previously assigned.[10] No $\nu_{C=C}$ was observed in dilute solution.

[b] Liquid film spectrum: $\nu_{C=C}$ 1640 cm^{-1} but *very* weak (private communication from Dr. Wells).

[c] Data from B. G. Cordiner, M. R. Vegar and R. J. Wells, *Tetrahedron Letters* 2285 (1970)

[d] Vinyl proton.

[e] Multiplet $W_{\frac{1}{2}}$ 9 Hz.

[f] ν (Hexane) 1678 and 1622 cm^{-1}.

EXPERIMENTAL

All m.ps are uncorrected. Routine IR spectra of liquid films and nujol mulls were recorded on a Unicam SP 200 spectrophotometer and quantitative spectra on a Perkin-Elmer Model 225. UV spectra, measured on a Unicam SP 800, refer to solutions in EtOH, unless otherwise stated. NMR spectra were obtained on the Varian T 60 instrument using TMS as internal standard. GLC was carried out on Pye Argon and Perkin-Elmer F 11 gas chromatographs. High resolution mass spectra were recorded on the AEI MS 12 mass spectrometer.

Bicyclo[5.3.1]*undecan*-8,11-*dione* (V). The literature method[5] gave a 75% yield of the dione, b.p. 108–110°/0·5 mm $\nu_{CO}^{CCl_4}$ 1729, 1704 cm^{-1}: ν_{CO}^{hexane} 1732, 1708 cm^{-1}.

Bicyclo[5.3.1]*undecan*-8-*ol*-11-*one toluene*-p-*sulphonate* (VII). The literature method[5] gave a 48% yield of product m.p. 118° (EtOH), NMR 5·4 τ (1 × H: 5-line signal, $W_{\frac{1}{2}}$ 17 Hz).

Bicyclo[5.3.1]*undec*-7-*en*-11-*one* (VIII). To a warm solution of the keto-tosylate (VII) (2·64 g, *i.e.* 7·8 m.mole) in dry EtOH (50 ml) was added, with stirring under N_2, 8 ml of 1·04 M ethanolic NaOEt (*i.e.* 8·2 m.mole of NaOEt). After 5 min sodium *p*-toluene sulphonate was precipitated. The solution was stirred for 14 hr at room temp, when the TLC showed no starting material remained. The mixture was poured into 50 ml of ice-water, extracted with ether and the extract washed with aq. $NaHCO_3$, then brine, dried and evaporated to give 1·23 g (96%) of a clear oil, b.p. 83–4°/2 mm. TLC showed a single spot, but GLC revealed two peaks in the ratio 98:2. Further purification was not achieved. (Found: C, 80·5: H, 9·88. $C_{11}H_{16}O$ requires C, 80·44: H, 9·83%), *m/e* 164. Retention index 1445 on 5% APL and 1870 on 5% QF1. The spectroscopic data (see Table) are in accord with structure VIII, and it yielded an oxime, identical with that previously reported (see Part 1).

Using the KOH-dioxane conditions,[5] the keto-tosylate yielded the same products (GLC).

Equilibration. The above mixture (98:2) of enones (6 mg) in 2 ml AcOH was treated with 0·5 ml conc. HCl and boiled under reflux for 60 hr. The solution was flooded with water, neutralized, and the organic material, in ether, was washed with brine, dried and evaporated. The residue showed the same two GLC peaks in the ration 75:25. These were too close to permit isolation of the second product, but the NMR of the crude sample showed in addition to the vinyl absorption *cf.* 3·85 τ, an unresolved multiplet centred at 4·3 τ which is ascribed to IX.

8-*Acetoxybicyclo*[5.3.1]*undec*-7-*en*-11-*one* (XII). The diketone (V) (360 mg), isopropenyl acetate (10 ml) and toluene-*p*-sulphonic acid (5 mg) were refluxed together for 114 hr. Most of the volatiles were removed *in vacuo*, the residue was taken up in ether, washed with aq. $NaHCO_3$, then brine, dried and concentrated. From the residue (304 mg), containing *ca.* 20% starting material (TLC) the acetate was isolated by prep. plate chromatography (Found: C, 69·94: H, 8·18. $C_{13}H_{18}O_3$ requires C, 70·24: H, 8·16%): λ_{max} 239 (ε 9,000): *m/e* 222: $\nu_{CO}^{CCl_4}$ 1758 and 1704 cm^{-1}.

Treatment of diketone V *with diazomethane*. An excess of ethereal CH_2N_2 was added to the diketone (2·121 g.) in dry MeOH (40 ml). The solution was allowed to stand 1 hr at 0° and then at room temp overnight, when GLC analysis showed little starting material and two products, A and B in the ratio 60:40. After filtration through 'celite' the solvent was removed under vacuum to yield 2·13 g of residue. Fractional distillation effected partial separation, giving two enriched fractions, (a) b.p. 78–80°/0·15 mm and (b) b.p. 80–84°/0·15 mm. The former yielded A by prep. TLC and the latter yielded B by column chromatography on alumina.

Compound A ($b_{0\cdot15}$ 79–80°, n_D^{20} 1·5103) showed $\nu_{C=O}$ 1720 and 1690 cm^{-1}, NMR 8·84 τ (3xH, s). This is consistent with that published for 7-methylbicyclo[5.3.1]undecan-8-11-dione[5] (XIV). Compound B, m.p. 73° (40–60 petrol). (Found: C, 74·00: H, 9·14. $C_{12}H_{18}O_2$ requires C, 74·19: H, 9·34%), showed λ_{max} 269 nm (ε = 8,660): NMR 6·26 τ (3H s): $\nu_{C=O}$ 1685 cm^{-1}, $\nu_{C=C}$ 1643 cm^{-1} and *m/e* 194, consistent with formulation of B as 8-methoxybicyclo[5.3.1]-undec-7-ene-11-one (XV).

8,8-*Dimethoxybicyclo*[5.3.1]*undecan*-11-*one* (XIII). A solution of the diketone V (10·8 g), 2,2-dimethoxypropane (7·28 g) MeOH (50 ml) and p-TsOH (30 mg) was stirred at room temp for 50 hr. The solution was neutralised with a methanolic solution of NaOMe and concentrated under reduced pressure. Work-up yielded 12·2 g residue, which recrystallized from 40–60 petrol to give 10·2 (75%) of a crystalline product, m.p. 66–67°: (Found C, 69·21: H, 9·94. $C_{13}H_{22}O_3$ requires C, 68·99: H, 9·80%) λ_{max} 293 nm (ε 20): NMR 6·83 τ (6H s): $\nu_{C=C}^{CCl_4}$ 1703 cm^{-1}: *m/e* 226.

Pyrolysis of ketal (XIII). (a) A solution of the ketal, (11·32 g) and p-TsOH (10 mg) in toluene (50 ml) was refluxed for 2 hr and the solvent distilled out, being replaced with fresh toluene, until 50 ml has been collected. The solution was refluxed for a further 2 hr when the solvent was removed under vacuum. The residue, dissolved in ether, was washed with aq. $NaHCO_3$ then brine, dried and concentrated to an oil, which consisted of the enol ethers XV and XVI in the ratio 55:45 (GLC).

(b) A solution of the ketal (2·26 g) was treated with NaOMe (from 46 mg Na) in 5 ml anhyd. MeOH. The solution was distilled to dryness at atmospheric pressure, then heated at 100°/15 mm for 1 hr, and the product finally distilled out, b.p. 96–102°/0·3 mm. Yield 1·69 g. GLC showed the presence of three compounds (90:8:2) of which the major was isolated by column chromatagraphy on alumina and was identical with XV. The other two were identified by GLC, as XVI and XIII respectively but could not be purified. On standing for several hr, unprotected, the former had decomposed to diketone (V).

Bicyclo[5.3.1]*undecan*-11-*ol*-8-*one* (XVII). The enol ether mixture (6·5 g) prepared by method (a) above, dissolved in anhyd. ether (50 ml) was added to a stirred suspension of LAH (1·27 g) in anhyd. ether (50 ml). The mixture was stirred at room temp for 19 hr, excess hydride destroyed with water (10 ml) and the solution extracted with ether. The extract was washed with brine, dried and evaporated yielding 5·05 g of residue which was dissolved in dioxan (50 ml) added to aqueous oxalic acid (20 ml of a 1M aqueous solution) and the solution stirred at room temp for 23 hr. Work-up yielded 4·85 g of an oil. A 2·6 g portion of this residue was chromatographed on 80 g of silica, when 2·425 g of GLC pure ketol was obtained as a low melting volatile solid, b.p. ~ 100 /0·7 mm. (Found: C, 72·20: H, 9·96. $C_{11}H_{18}O_2$ requires C, 72·49: H, 9·95%) λ_{max} 282 nm (ε 38·5). ν_{OH} 3625 cm^{-1}, $\nu_{C=O}$ 1713 cm^{-1}: *m/e* 182.

Bicyclo[5.3.1]*undec*-7(11)-*en*-8-*one* (XIX). The ketol (480 mg) in anhyd. pyridine (4 ml) was added, with stirring at 0° to a solution of MesylCl (0·3 ml) in anhyd pyridine (2 ml). When addition was complete (15 min), the mixture was stirred at room temp. for 4 hr, then poured into ice (15 g) and extracted with benzene. The extract was washed with aq. HCl, aq. Na_2CO_3 and finally brine, dried and concentrated under vacuum, giving 550 mg (80%) of an oily mesylate which crystallized m.p. 60–61° from benzene/40–60 petrol. (Found: C, 55·16: H, 7·48. $C_{12}H_{20}O_4S$ requires C, 55·33: H, 7·74%), *m/e* 260. NMR 4·83 τ (1 × H q: $W_{\frac{1}{2}}$ 14 Hz): ν 1715, 1367, 1178 cm^{-1}.

To the mesylate (3·86 g) in benzene (70 ml) was added, dropwise, with stirring, 1,5-diazabicyclo[4.3.0]non-5-ene (DBN) (4·1 g) in benzene (80 ml). After 15 min a white solid began to be precipitated and when addition was complete (30 min) the mixture was stirred for a further hour. The solution was filtered and the solvent removed *in vacuo*. On addition of ether, further precipitation took place. The ethereal solution was washed with aq. HCl, then brine, dried (Mg SO_4) and evaporated to an oil (1·26 g) which was purified either by prep. plate chromatography (HF 254 silica: 30% EtOAc/petrol; R_f 0·5) or by flash distillation *in vacuo* (n_D^{25} 1·5356). The resulting pure enone showed one peak on GLC (R_I 1888 on 5% QF1). (Found: C, 80·62: H, 9·62. $C_{11}H_{16}O$ requires C, 80·44: H, 9·82%), *m/e* 164: NMR 3·15 τ (1 × H: d, J = 4·5 Hz). For other spectral data, see Table. On brief standing it deposited a white amorphous powder: *m/e* (Parent) 348 Calc. for $C_{22}H_{32}O_2$ 328.

11-*Phenyl bicyclo*[5.3.1]*undec*-7(11)-*en*-8-*one* (XXI). (a) To the enol mixture (598 mg: *i.e.* 3·1 m.mole) in anhyd. diethyl ether (10 ml) was added a solution of PhMgBr in anhyd. ether (equivalent to 3·5 m.moles). The mixture was allowed to stir at room temp. for 45 min, poured into an ice-ammonium chloride-water slurry, extracted with ether, washed with brine and concentrated. The residue (605 mg) was chromatagraphed on silica. Elution with benzene-petrol (1:1) gave 421 mg of crystalline material. Recrystallization from 40–60 petrol gave the intermediate (XX), m.p. 154–6°. (Found: C, 79·23; H, 8·69. $C_{18}H_{24}O_2$ requires C, 79·37: H, 8·88%). *m/e* 272. $\nu_{OH}^{CCl_4}$ 3600 cm^{-1}, $\nu_{C=C}^{CCl_4}$ 1673 cm^{-1}, NMR 2·3–2·8 τ (5H): 5·20 τ (1H) triplet (J = 4 Hz), 6·40 τ (3H) singlet.

(b) The hydroxy enol-ether (XX) above, (63·4 mg), dissolved in MeOH (10 ml) containing *p*-TsOH (2 mg) and water (0·5 ml) was refluxed in an atmosphere of N_2 for 20 hr. Solvent was removed, ether added, and the ethereal solution washed with aq. Na_2CO_3, then brine, dried and concentrated, to yield 56 mg of yellow oil. The product (40 mg), isolated by prep. TLC had m.p. 92–92·5 (40–60 petrol). (Found: C, 84·93: H, 8·45. $C_{17}H_{20}O$ requires C, 84·96: H, 8·39%), *m/e* 240, λ_{max} 308 nm (ε = 13,500): NMR 2·4–2·8 τ (5H: mult.), $\nu_{C=O}^{hexane}$ 1660 and $\nu_{C=C}$ 1652 cm^{-1}.

Attempted Michael-addition. Diethyl malonate (150 mg) was treated with ethanolic NaOEt (from 20 mg Na in 8 ml anhyd. EtOH) and cooled to 0°. To this solution, 150 mg of the enone (VIII) in 2 ml anhyd. EtOH was added with stirring over 5 min., and stirring continued at room temperature for 48 hr. GLC analysis of an aliquot showed no new component. The mixture was then refluxed for 70 hr., cooled, flooded with water, extracted with ether, and the extract washed with brine, dried and evaporated. The residue (160 mg) showed (GLC) traces of diethyl malonate and the enones VIII and IX in the ratio 99:1, but no other product at > 0·5% concentration.

Epoxidation. A solution of the enone (30 mg) in 8% NaOH aq (1 ml), 15% H_2O_2 (1 ml) and EtOH (1 ml) was warmed (steam bath) for two min and stirred at room temperature for 3 days. When it was flooded with ice water (10 ml), and worked up as usual, it gave 22 mg of material, shown (GLC) to contain starting enone (50%) and a new product (50%) which was isolated by prep. plate chromatography. The oil epoxide (XXV), b.p. 70–75°/0·25 mm, (Found: C, 73·09; H, 8·89. $C_{11}H_{16}O_2$ requires C, 73·30: H, 8·95%) had R_I 2130 on 5% QF1 and 1545 on 5% APL: *m/e* 180: $\nu_{CO}^{CCl_4}$ 1732 cm^{-1}: NMR 6·68 τ (1 × H: br. singlet).

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