Free Radical Addition of Isobutyric Acid to α,ω-Diolefins. A New Route to α,α,α′,α′-Tetramethylalkanedioic Acids

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2,2,9,9-Tetramethylsebacic and 2,2,8,8-tetramethylazelaic acids have been synthesized in reasonable yields (55% in the case of the former) by the free radical addition of isobutyric acid to hexa-1,5-diene and penta-1,4-diene respectively using di-t-butyl peroxide as initiator. Extension of this method to hepta-1,6-diene and octa-1,7-diene led to the production of new cyclic compounds. *cis*- and *trans*-2,2-Dimethyl-3-(2-methylcyclopentyl)propionic acids were obtained from hepta-1,6-diene and *cis*- and *trans*-2,2-dimethyl-3-(2-methylcyclohexyl)propionic acids and 2,2-dimethyl-3-cycloheptylpropionic acid from octa-1,7-diene. The mechanisms of these reactions are discussed. Some attempted additions of isobutyric acid to methylene cycloalkanes containing five-, six-, and seven-membered rings are described.

THE main aim of our studies on the addition of isobutyric acid to α,ω-diolefins was to develop a simple synthetic route to α,α,α′,α′-tetramethylalkanedioic acids. These compounds are useful intermediates in the synthesis of stable lubricants for aero gas turbine engines. Although extensive studies [1-6] have been made on the free radical addition of acids to mono-olefins, relatively

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[1] J. C. Allen, J. I. G. Cadogan, and D. H. Hey, *J. Chem. Soc.*, 1965, 1918.

[2] J. I. G. Cadogan, D. H. Hey, and S. H. Ong, *J. Chem. Soc.*, 1965, 1832.

[3] J. I. G. Cadogan, D. H. Hey, and J. T. Sharp, *J. Chem. Soc.* (*C*), 1966, 1743; *J. Chem. Soc.* (*B*), 1967, 803.

[4] A. D. Petrov, G. I. Nikishin, and Yu. N. Ogibin, *Doklady Akad. Nauk S.S.S.R.*, 1960, **131**, 580 (*Chem. Abs.*, 1960, **54**, 16,381); *Trudy Khim. i khim. Tekhnol.*, 1961, **4**, 123 (*Chem. Abs.*, 1961, **55**, 27,052).

[5] A. D. Petrov, G. I. Nikishin, V. D. Vorobiev, and Yu. N. Ogibin, *Chem. Abs.*, 1963, **59**, 11,234.

[6] H.-H. Vogel, *Synthesis*, 1970, 99.

Table 1

Addition of isobutyric acid to α,ω-diolefins using Bu^tOOBu^t as initiator (mole ratio acid : diolefin : initiator 100 : 1 : 0·8)

α,ω-Diolefin	Temp. (°C)	Diolefin conversion (%)	Products (% yield based on diolefin converted)
Penta-1,4-diene	145	44	(I) 2,2-Dimethylhept-6-enoic acid (20%)
			(II) 2,2,8,8-Tetramethylazelaic acid (24%)
Hexa-1,5-diene	140	82	(III) 2,2-Dimethyloct-7-enoic acid (42%)
			(IV) 2,2-Dimethyl-3-cyclopentylpropionic acid (3%)
			(V) 2,2,9,9-Tetramethylsebacic acid (28%)
Hepta-1,6-diene	140	95	(VII) *cis*-2,2-Dimethyl-3-(2-methylcyclopentyl)-propionic acid (22%)
			(VIII) *trans*-2,2-Dimethyl-3-(2-methylcyclopentyl)-propionic acid (40%)
Octa-1,7-diene *	140	98	(X) *trans*-2,2-Dimethyl-3-(2-methylcyclohexyl)-propionoic acid (9%)
			(XI) *cis*-2,2-Dimethyl-3-(2-methylcyclohexyl)-propionic acid (17%)
			(XIII) 2,2-Dimethyl-3-cycloheptylpropionic acid (6%)

* Olefin : initiator ratio 1 : 0·4.

little work [6] has been carried out with diolefins. Generally organic peroxides are efficient initiators of such reactions.

Table 2

Addition of isobutyric acid to hexa-1,5-diene

Run no.	Reactant concentrations (molar ratios) Isobutyric acid	Hexa-1,5-diene	t-Butyl peroxide	Temp. (°C)	Diene converted (%)	Yields (%) based on diene converted Monoadduct	Bisadduct	Comments
1	100	1	0·8	140	82	42	28·5	
2	100	1	0·8	148	86	38·5	38	
3	100	1	0·8	160	97·5	21	56	25% (mol) Acid as potassium salt
4	25	1	0·8	140	100	23	35	
5 *	100	25	1 †	180	N.d.	15	Trace	
6 *	100	4	0·8	130	85	42	25	Reaction chain length ≈ 2

N.d. = Not determined.

* Reaction carried out in glass pressure bottle with all reactants present initially. † Cumyl hydroperoxide as initiator.

In this paper we describe our studies with a series of α,ω-diolefins culminating in the successful syntheses of 2,2,8,8-tetramethylazelaic and 2,2,9,9-tetramethylsebacic acids. With certain diolefins, free radical cyclizations leading to new compounds were observed and these are described.

RESULTS

High dilution techniques were used and the molar ratio acid: diolefin : initiator was usually *ca.* 100 : 1 : 0·4—0·8.

The initiator, dissolved in isobutyric acid, was added to the reaction vessel at atmospheric pressure by means of a micropump over 3—24 h, normally 4—6 h. Temperatures varied from ambient to 180° depending on the initiator used. After the reaction was complete, the excess of isobutyric acid and unchanged diolefin was distilled off. The residual acids either as free acids or methyl esters were examined by g.l.c.: specific components of interest were isolated using preparative g.l.c. and identified by their n.m.r., i.r., and mass spectra. When either very volatile diolefins were used or elevated temperatures (>150°) were required, reactions were carried out at moderate pressures (maximum 17 bar) in glass pressure bottles. Data obtained for additions to a range of α,ω-diolefins are given in Table 1.

For hexa-1,5-diene, the effect on yields of varying the molar proportions of the reactants and the temperature is shown in Table 2.

Additions of isobutyric acid to three methylenecycloalkanes, methylenecyclopentane, 1-methyl-2-methylenecyclohexane, and methylenecycloheptane were attempted in order to synthesize authentic samples of 2,2-dimethyl-3-cyclopentylpropionic, 2,2-dimethyl-3-(2-methylcyclohexyl)propionic, and 2,2-dimethyl-3-cycloheptylpropionic acids respectively. These were required for comparison with cyclic products from the reactions of isobutyric acid with hexa-1,5-diene and octa-1,7-diene. The latter two methylenecycloalkanes were synthesized *via* Wittig reactions [7] from the corresponding ketones in yields of 37 and 59% respectively. The results of the addition reactions are shown in Table 3.

Product Analysis.—The crude products were converted into their methyl esters and the latter were distilled and separated into individual components by preparative g.l.c. The structure elucidation of the various esters was based mainly on mass and n.m.r. spectral analysis, and the major details are given in Tables 4 and 5. I.r. spectra were in agreement with the assignments. The products listed do not include tetramethylsuccinic anhydride and tetramethylsuccinic acid found to be present in all free radical additions of isobutyric acid to olefins.

(a) *Penta-1,4-diene.* The major components of the pro-

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[7] A. Maercker, *Org. Reactions*, 1965, **14**, 395.

duct were identified as the expected mono- (I) and bis-adduct (II) by examination of the derived esters. The presence of the $C(Me)_2CO_2Me$ group in both esters was indicated by the ester methyl singlet at τ *ca.* 6·4 and the *gem*-dimethyl singlet at τ *ca.* 8·9, and the prominent fragment ion at *m/e* 102 in the mass spectra. The presence of terminal unsaturation in (I) was shown by the multiplets at τ 4·20, 4·91, and 5·08, and also by the i.r. spectrum (bands at 3·2, 6·05, and 10·95 μm). The parent ions at *m/e* 170 and 272 in the mass spectra were consistent with structures (I) and (II).

TABLE 3

Addition of isobutyric acid to methylenecycloalkanes using Bu^tOOBu^t as initiator (mole ratio acid : olefin : initiator 30 : 1 : 1)

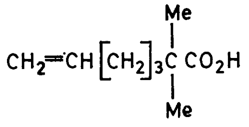
Olefin	Temp. (°C)		Main products
Methylenecyclopentane	145	(IV)	2,2-Dimethyl-3-cyclopentylpropionic acid (2 parts)
		(VI)	2,2-Dimethyl-3-cyclopentylidenepropionic acid (1 part)
1-Methyl-2-methylene cyclohexane	140		Complex mixture. No 2,2-dimethyl-3-(2-methylcyclohexyl)-propionic acid present
Methylenecycloheptane	140	(XIV)	2,2-Dimethyl-3-(cyclohept-1-enyl)propionic acid (10%)

TABLE 4

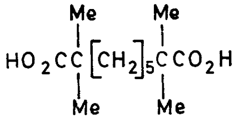
Significant peaks in mass spectra of addition products (as methyl esters)

Diene	Products	*m/e*	Assignment
Penta-1,4-diene	(I)	170	$C_{10}H_{18}O_2$
		102	$C_5H_{10}O_2$
	(II)	272	$C_{15}H_{28}O_4$
		102	$C_5H_{10}O_2$
Hexa-1,5-diene	(III), (IV)	184	$C_{11}H_{20}O_2$
		102	$C_5H_{10}O_2$
	(V)	286	$C_{16}H_{30}O_4$
		102	$C_5H_{10}O_2$
Methylenecyclopentane	(VI)	182	$C_{11}H_{18}O_2$
Hepta-1,6-diene	(VII), (VIII)	198	$C_{12}H_{22}O_2$
Octa-1,7-diene	(X), (XI), (XIII)	212	$C_{13}H_{24}O_2$
		155	$C_9H_{15}O_2$
		102	$C_5H_{10}O_2$
Methylenecycloheptane	(XIV)	210	$C_{13}H_{22}O_2$

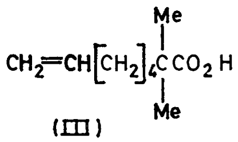
(b) *Hexa*-1,5-*diene*. The major components (III)—(V) were assigned the structures shown. The presence of the $C(Me)_2CO_2Me$ group in all three after esterification was confirmed by the mass and n.m.r. spectra as for the penta-1,4-diene adducts. The presence of terminal unsaturation in (III) was indicated by the multiplets at τ 4·25, 4·95, and 5·13 and also by the i.r. spectrum (bands at 3·2, 6·05, and 10·95 μm). No evidence for unsaturation or for additional methyl groups was found in either the n.m.r. or i.r. spectra of components (IV) and (V). This and the mass spectral evidence is consistent with the mono- and bis-adduct structures shown.


$CH_2{=}CH[CH_2]_3\underset{Me}{\overset{Me}{C}}CO_2H$

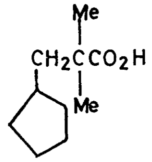
(I)


$HO_2C\underset{Me}{\overset{Me}{C}}[CH_2]_5\underset{Me}{\overset{Me}{C}}CO_2H$

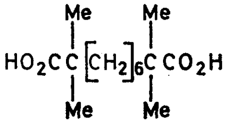
(II)


$CH_2{=}CH[CH_2]_4\underset{Me}{\overset{Me}{C}}CO_2H$

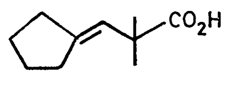
(III)



(IV)


$HO_2C\underset{Me}{\overset{Me}{C}}[CH_2]_6\underset{Me}{\overset{Me}{C}}CO_2H$

(V)



(VI)

In an attempt to synthesize compound (IV) independently, the free radical addition of isobutyric acid to methylenecyclopentane was carried out. The product mixture contained two major components isolated as their methyl

TABLE 5

N.m.r. spectral details of addition products (as methyl esters)

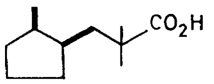
		τ Values				
Olefin	Product	CH=CH$_2$	CO$_2$Me	CH$_2$	>CMe$_2$	CHMe
Penta-1,4-diene	(I)	4·20(m) 4·91(m) 5·08(m)	6·35(s)	8·55	8·88	
	(II)		6·38(s)	8·74	8·88	
Hexa-1,5-diene	(III)	4·25(m) 4·95(m) 5·13(m)	6·36(s)	8·00	8·87(s)	
	(IV)		6·42(s)	8·40	8·88(s)	
	(V)		6·33(s)	8·57 8·81	8·84(s)	
Methylenecyclopentane	(VI)	4·55(s) †	6·38(s)	7·60	8·84(s)	
Hepta-1,6-diene	(VII)		6·37(s)	8·43	8·90(d)	9·08(d)
	(VIII)		6·38(s)	8·46	8·88(s)	9·23(d)
Octa-1,7-diene	(X)		6·36(s)	8·56	8·83(s)	9·05(s)
	(XI)		6·36(s)	8·56	8·83(s)	9·05(d)
	(XIII)		6·36(s)	8·48	8·83(s)	
Methylenecycloheptane	(XIV)	4·44(t) ‡	6·39(s)	7·78	8·85(s)	

s = Singlet, d = doublet, t = triplet, m = multiplet.

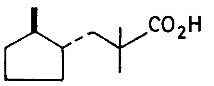
* Centre of broad band. † $CMe_2CH{=}C<$. ‡ $CH_2CH{=}C<$.

esters by preparative g.l.c. The mass and n.m.r. spectra of the component of shorter retention time on a polar g.l.c. column were identical to those of the second main component of the hexa-1,5-diene adduct, thus confirming the assigned structure (IV). The mass spectrum of the component of longer retention time indicated a parent ion at *m/e* 182 (Table 4) and the n.m.r. details are shown in Table 5. The latter evidence, especially the olefinic singlet at τ 4·55, suggests that the acid giving rise to this component has the structure (VI).

(c) *Hepta*-1,6-*diene*. The major components of the product mixture in this case were two monoadducts of molecular formula $C_{12}H_{22}O_2$ (methyl ester). These were assigned as the methyl esters of (VII) and (VIII), *viz.*,



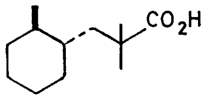
(VII)



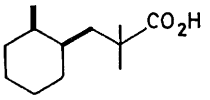
(VIII)

methyl *cis*- and *trans*-2,2-dimethyl-3-(2-methylcyclopentyl)-propionates. No evidence for unsaturation was detected. The n.m.r. data is consistent with (VII), the isomer of shorter g.l.c. retention time, being the *cis*-isomer, since the steric hindrance to free rotation of the >CMe$_2$ group arising from its proximity to the ring methyl group could result in a non-equivalence of the *gem*-dimethyl groups. Free rotation of the *gem*-dimethyl groups is possible in the *trans*-isomer and the n.m.r. signal is a singlet. The ring methyl group was indicated by doublets at τ 9·08 and 9·23 respectively.

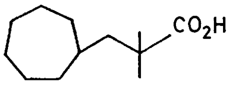
(d) *Octa*-1,7-*diene*. The monoadduct fraction consisted of the methyl esters of five main components, (IX)—(XIII), the percentage yields on converted diene being as follows: (IX) 5, (X) 9, (XI) 17, (XII) 4, and (XIII) 6%. The structures



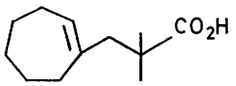
(X)



(XI)



(XIII)



(XIV)

of (X), (XI), and (XIII) are as shown but definite structures could not be assigned to (IX) and (XII). Neither appeared to be the acyclic monoadduct, methyl 2,2-dimethyldec-9-enoate. The mass spectral parent ions at *m/e* 212 and the absence of unsaturation in the n.m.r. and i.r. spectra indicate that (X), (XI), and (XIII) are cyclic. Ring methyl groups gave rise to peaks at τ 9·05 in the case of (X) and (XI) (broad singlet and a doublet, respectively). The n.m.r. spectrum of (XI) was considerably sharper than that of (X) which is consistent with the (XI) being the *cis*-isomer, *i.e.* the isomer with the somewhat less rigid structure. No methyl resonance above τ 9·0 was detected in the n.m.r. spectrum of (XIII) which is in accord with the assigned structure.

In an attempt to synthesize compounds (X), (XI), and (XIII) independently, the free radical addition of isobutyric acid to 1-methyl-2-methylenecyclohexane and methylenecycloheptane was carried out. A complex mixture of products was obtained from the former, none of which corresponded to the cyclohexyl derivatives (X) and (XI). The mixture was not further examined. The product from methylenecycloheptane contained a major component isolated as its methyl ester. Both the mass (Table 4) and n.m.r. (Table 5) spectra suggest that the acid giving this component has the structure (XIV), in particular on the evidence provided by the olefinic triplet at τ 4·44. Hydrogenation of a sample of the ester from (XIV) over Adam's catalyst gave an ester with mass and n.m.r. spectra identical with those of the ester from (XIII).

DISCUSSION

Additions to Penta-1,4-*diene and Hexa*-1,5-*diene*.—With penta-1,4-diene and hexa-1,5-diene, the reactions went smoothly to yield 2,2,8,8-tetramethylazelaic and 2,2,9,9-tetramethylsebacic acids respectively. In all experiments mixtures of mono- and bis-adduct were obtained; in a process for the production of α,α,α′,α′-tetramethylalkanedioic acids, the former would be recycled to produce more bisadduct. Both diolefins gave similar yields when reacted under similar conditions.

Using the conditions, 140°, Bu^tOOBu^t as initiator, acid : diene : initiator molar ratio of 100 : 1 : 0·8, addition to hexa-1,5-diene gave a yield of almost 30% bisadduct and >40% monoadduct at 82% diolefin conversion. Increasing the reaction temperature by adding potassium isobutyrate had a beneficial effect (runs 1—3, Table 2) and at 160°, yields of 56% bis- and 21% mono-adduct were obtained at almost quantitative diolefin conversion; with recycling, this represents a potential 70% yield of 2,2,9,9-tetramethylsebacic acid from a simple one-step reaction.

The high molar ratio (100 : 1) of acid : diolefin used in these experiments was chosen to avoid telomerization of the diolefin. Reduction of the isobutyric acid concentration by a further factor of four did not significantly reduce the yields (runs 1 and 4, Table 2). A single high temperature experiment (run 5) at 180° using cumyl hydroperoxide as initiator gave poor results. In run 6, the proportion of initiator to diolefin was decreased and a value for the chain length (calculated from the amount of adduct formed per initiating radical) of two was found thus confirming that the addition is a chain reaction, if a very modest one.

In all experiments, small amounts of non-volatile residues were found; these are probably telomeric materials or carboxylic acid polymers formed in a similar manner to that described by Trecker and Foote.[8] In the case of hexa-1,5-diene, a minor component (3%) of the reaction products was a cyclic compound (IV). The formation of this compound is of particular interest in view of the fairly extensive evidence [9-11] that cycliza-

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[8] D. J. Trecker and R. S. Foote, *J. Org. Chem.*, 1968, **33**, 3527.

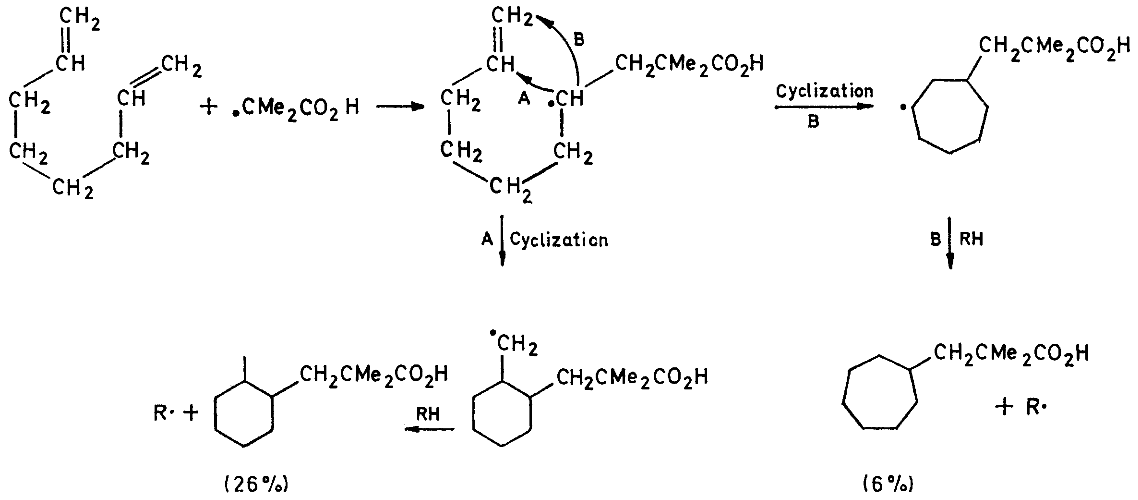
[9] N. O. Brace, *J. Org. Chem.*, 1967, **82**, 2711.

[10] C. Walling and M. S. Pearson, *J. Amer. Chem. Soc.*, 1964, **86**, 2272.

[11] M. Julia and F. Le Goffic, *Bull. Soc. chim. France*, 1965, 1550.

tion of pent-4-enyl radicals to form cyclopentyl derivatives does not occur.

*Additions to Hepta-*1,6-*diene and Octa-*1,7-*diene.*—From octa-1,7-diene no significant amount of the bis-adduct, 2,2,11,11-tetramethyldodecanedioic acid, was found and a complex mixture of products was obtained. The major identifiable components of the reaction corresponded to the cyclic monoadducts, *cis*- and *trans*-2,2-dimethyl-3-(2-methylcyclohexyl)propionic acid (XI) and (X) respectively. We consider that these isomers were formed by Path A in the Scheme. Much smaller amounts of the cycloheptyl compound, 2,2-dimethyl-3-cycloheptylpropionic acid, formed by intramolecular addition to the terminal carbon (Path B) were also observed. The compounds formed are new and this route represents an interesting method for making alicyclic α,α-dimethylalkanoic acids.



SCHEME

Although many free radical cyclizations are known this particular example has not been reported previously. Cyclizations to produce cyclohexyl derivatives have been observed by several workers;[2,12] these studies were carried out in systems yielding free radicals of type (XV)

$$R^1R^2C{=}CH[CH_2]_3\dot{C}R^3R^4$$

(XV)

where R^1R^2 = H, Me and R^3R^4 = CN, CO_2Et. In cases where the radical site is not substituted, *i.e.*, $R^3 = R^4 = H$, cyclopentyl derivatives are formed.[9,10] The reasons for cyclohexyl *versus* cyclopentyl ring formation in these systems have been considered in depth, notably by Julia and his co-workers.[12,13] A significant factor is resonance stabilization of the radical site which normally leads to the thermodynamically favoured cyclohexyl ring formation. In the 1,7-diolefin system there is a fairly good orbital overlap between the radical and C(6) which would promote the formation of substituted cyclohexyl products at the expense of cycloheptyl derivatives.

Our results with hepta-1,6-diene in which a fairly good overlap occurs between the radical and both C(5) and C(6)[13] are in agreement with trends observed by the other workers mentioned although the specific radical encountered in our studies had not been investigated hitherto. It corresponds to a radical of type (XV) with $R^1 = R^2 = H$ and $R^3 = H$, $R^4 = CH_2CMe_2CO_2H$, *i.e.* a non resonance-stabilized radical which might be expected to yield cyclopentyl derivatives. The products found were mainly cyclopentyl compounds and their formation can be accounted for by a similar scheme to that shown (Path A) for octa-1,7-diene. The main product, 2,2-dimethyl-3-(2-methylcyclopentyl)propionic acid can exist as *cis*- and *trans*-isomers and we found a ratio of 2 : 1 in favour of the *trans*-isomer. A study of molecular models showed that for the *cis*-isomer only, there is considerable steric interaction between the methyl and the $CH_2CMe_2CO_2H$ groups in adjacent positions on the cyclopentane ring. No significant amount of cyclohexyl derivatives was found.

Additions to Methylenecycloalkanes.—In the additions to methylenecyclopentane and methylenecycloheptane, the formation of the unsaturated cyclic products, 2,2-dimethyl-3-cyclopentylidene- and 2,2-dimethyl-3-(cyclohept-1-enyl)-propionic acids, respectively suggests that the intermediate adduct radicals (XVI) and (XVII) undergo disproportionation rather than hydrogen abstraction reactions. Abstraction of the tertiary hydrogen atom from isobutyric acid by these bulky, relatively stable radicals would be sterically hindered. In the case of 1-methyl-2-methylenecyclohexane, the corresponding radical (XVIII) is even more sterically restricted and no compound corresponding to either a disproportionation or hydrogen abstraction product was found.

In general terms it seems that the course of the addition of isobutyric acid to α,ω-diolefins is critically dependent

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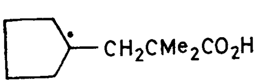
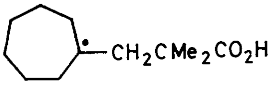
[12] M. Julia and M. Maumy, *Bull. Soc. chim. France*, 1968. 1603.

[13] M. Julia, *Pure Appl. Chem.*, 1967, **15**, 167.

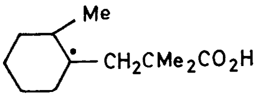
on the length of the methylene bridge between the double bond sites, *i.e.*, the value of n in $CH_2=CH[CH_2]_n$-$CH=CH_2$. The known or expected products for a series of α,ω-diolefins are: $n = 0$, expected to give 1,4-addition products or unsaturated dimer acids by dimerization of allylic free radicals produced by mono-addition; $n = 1$, yields α,α,α′,α′-tetramethylalkanedioic acids; $n = 2$, yields α,α,α′,α′-tetramethylalkanedioic acids; $n = 3$, yields cyclic products; $n = 4$, yields cyclic products; and $n > 5$, expected to yield mainly α,α,α′,α′-tetramethylalkanedioic acids. The main limitation of this synthetic route to α,α,α′,α′-tetramethylalkanedioic acids is heavy consumption of the free radical initiator.



(XVI)



(XVII)



(XVIII)

EXPERIMENTAL

I.r. spectra were measured on a Perkin-Elmer Infracord spectrometer. All the products, after methylation with diazomethane, exhibited characteristic C=O and C–O stretching absorption frequencies at 1720 and in the 1100—1250 cm^{-1} region respectively. Normal and integrated n.m.r. spectra were obtained on a modified Varian instrument (upgraded to 80 MHz) using carbon tetrachloride as solvent and tetramethylsilane as internal standard; integrations were satisfactory (±5%) and chemical shift data are given in Table 5. Mass spectra were determined on an AEI MS 12 instrument. G.l.c. of acidic products was carried out analytically on a Pye 104 gas chromatograph equipped with a 1·5 m × 6·5 mm column packed with 10% FFAP on 80—100 mesh Chromosorb W for the mono-adducts and a similar 0·5 m × 6·5 mm column for the bis-adducts. The methyl esters were also analysed on the former column but those obtained from octa-1,7-diene were analysed on a 46 m × 0·25 mm stainless steel capillary column coated with Apiezon L grease. Peak areas were determined by means of a Kent Chromolog integrator. Preparative g.l.c. was carried out on a Pye 105 model 15 instrument equipped with: (i) a 9·2 m × 9·5 mm column packed with 20% SE52 silicone gum on 60—72 mesh, acid-washed, siliconized Diatomite C; (ii) a 4·6 m × 9·5 mm column packed with 20% XE60 cyanosilicone gum on 60—80 mesh Chromosorb A; (iii) a 2·2 m × 9·5 mm column packed with 15% FFAP on 60—72 mesh, acid-washed siliconized Diatomite C; and (iv) a 9·2 m × 9·4 mm column packed with 20% diethylene glycol succinate polyester on 45—60 mesh Chromosorb A. Trapping of peaks was effected using packed trap techniques.

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