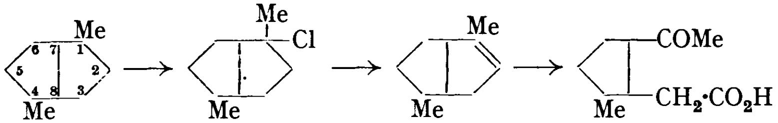
139. *Fused Carbon Rings. Part XI. The Action of Aluminium Chloride on Decalin.*

By R. L. Jones and R. P. Linstead.

Zelinski and Turowa-Pollak (*Ber.*, 1925, **58**, 1292) have shown that both hydrogen iodide at 300° and aluminium bromide at 100° react with decalin with the formation of lower-boiling hydrocarbons (compare Ross and Leather, *Analyst*, 1906, **31**, 289; Baedtker and Rambeck, *Bull. Soc. chim.*, 1924, **35**, 631). Using the latter reagent, they isolated in small amount a dicyclic saturated hydrocarbon, isomeric with decalin but boiling at 160—165°, which they suggested might be a dimethyl-0 : 3 : 3-*bicyclo*octane. Although this appeared reasonable, the evidence for it was slight and, as a general investigation of 0 : 3 : 3-*bicyclo*octanes was in progress, it was decided to reinvestigate this substance and compare its properties with those of authentic hydrocarbons of the series.

The rearrangement of decalin was conveniently effected by means of aluminium chloride at 130°. The isomerised material was accompanied by small amounts of low-boiling monocyclic hydrocarbons. Systematic fractionation yielded a peak fraction boiling at about 160° and resembling that obtained by Zelinski and Turowa-Pollak. This substance, which was isomeric with decalin, was unaffected by bromine, permanganate, sulphuric acid, or sulphur dioxide, and hence contained two stable rings. It was not dehydrogenated over a nickel catalyst at 300°. It is well known, particularly from Zelinski's work (*e.g.*, *Ber.*, 1911, **44**, 3124; 1912, **45**, 3678), that the capacity for dehydrogenation distinguishes *single cyclo*hexane from *cyclo*pentane rings. Two five-membered rings fused in the ortho-position, however, present a special case in which dehydrogenation is conceivable. It has now been proved experimentally that the 0 : 3 : 3-*bicyclo*octane system cannot be dehydrogenated (Barrett and Linstead, preceding paper *) and the resistance to dehydrogenation is therefore consistent with the dimethyl-0 : 3 : 3-*bicyclo*octane structure in the present instance.

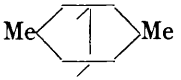
Chlorination of the isomerisation product (b. p. *ca* 160°) over iodine or iron gave a mixture, from which a *monochloro*-compound, $C_{10}H_{17}Cl$, was separated. This on treatment with boiling aniline yielded an unsaturated *hydrocarbon*, $C_{10}H_{16}$. When this was oxidised by means of potassium permanganate a mixture of acids was obtained, from which a small amount of a solid *keto-acid*, $C_{10}H_{16}O_3$, was isolated. This gave iodoform on treatment with iodine and caustic soda; hence the parent hydrocarbon contained a $CH_3{\cdot}C\colon$ group. One simple explanation for these facts is that the isomerisation product and the substances derived from it have the formulæ (I to IV) :



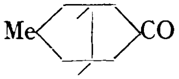
(I.) (II.) (III.) (IV.)

These substances are capable of existing in a number of stereoisomeric forms and the difficulty experienced in the isolation of pure compounds is easily understood.

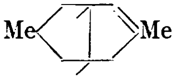
No authentic dimethyl-0 : 3 : 3-*bicyclo*octane was available for a comparison of physical properties with those of the isomerisation product. Experiments directed towards the synthesis of the 1 : 4-dimethyl derivative (I) were unpromising and we therefore turned to the much simpler preparation of the 2 : 5-isomeride (V). This was prepared from ethyl 4-methyl*cyclo*pentanone-2-carboxylate through *4-methyl*cyclo*pentane-1 : 2-diacetic acid* by the general method previously described (Linstead and Meade, J., 1934, 935; Barrett and Linstead, J., 1935, 436).



(V.)



(VI.)



(VII.)

The acid was cyclised to *5-methyl*-0 : 3 : 3-bicyclo*octan-2-one* (VI), and this converted through 2 : *5-dimethyl*-0 : 3 : 3-bicyclo*octan-2-ol* into 2 : *5-dimethyl*-Δ^1-0 : 3 : 3-bicyclo*octene* (VII). No special attempt was made to separate the *cis*- and the *trans*-isomeride, but as the cyclisation to the ketone (VI) took place at about 280°, the dicyclic compounds must have been essentially *cis*-. The physical properties of the *bicyclo*octene (VII) agreed fairly closely with those of the unsaturated hydrocarbon (? III) prepared from the isomerisation product of decalin (see table). Catalytic reduction of (VII) yielded *cis*-2 : *5-dimethyl*-0 : 3 : 3-bicyclo*octane* (V). The physical properties of this are compared below with those of the isomerisation products of decalin obtained by Zelinski and Turowa-Pollak and in the present work. There is a general agreement and an indication that the material from

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* Publication of the present work, which was completed in 1933, was postponed in order that this fact might be established.

decalin has the *trans*-configuration. This view would fall into line with the observation of Zelinski and Turowa-Pollak (*Ber.*, 1929, **62**, 1658) that *cis*-hydrindane is isomerised into the *trans*-modification by aluminium bromide.

Substance.	B. p.	$d_{4^\circ}^{20^\circ}$.	$n_D^{20^\circ}$.	$[R_L]_D$.
$C_{10}H_{16}$ from decalin	160°	0·863	1·466	43·66
$C_{10}H_{16}$ synthetic	167	0·865	1·465	43·65
$C_{10}H_{18}$ from decalin (Z., T.-P.)	160—165	0·832	1·449	44·50
$C_{10}H_{18}$ from decalin (J., L.)	158—163	0·832	1·448	44·44
$C_{10}H_{18}$ synthetic	166—167	0·838	1·450	44·25

The experimental figures have been corrected to 20°, where necessary, to assist comparison.

These synthetic and analytical results therefore support, although they do not prove, the correctness of Zelinski's view of the constitution of the isomerisation product of decalin. It is reasonable to suppose that the methyl groups would be symmetrically disposed, and of the three possible positions (1 : 4-, 2 : 5-, and 7 : 8-), the 1 : 4- is the most probable from the method of formation and the 7 : 8- is excluded by the formation of the keto-acid $C_{10}H_{16}O_3$. It is curious that no solid hydrocarbons of the 1 : 2 : 3-*bicyclo*octane series (compare Part X, preceding paper) were isolated during the rearrangements. Possibly this was due to the difference in experimental conditions. The three known isomerisations of *ortho*-dicyclic hydrocarbons by aluminium chloride (*cis*- ⟶ *trans*-decalin; decalin ⟶ dimethyl-0 : 3 : 3-*bicyclo*octane; 0 : 3 : 3-*bicyclo*octane ⟶ 1 : 2 : 3-*bicyclo*octane) can all be interpreted by the breaking of *one* bond of the dicyclic structure, that connecting a bridge with an α-carbon atom, followed by the migration of one hydrogen atom and the re-formation of a carbon-to-carbon bond.

The low-boiling products of the action of aluminium chloride on decalin were fractionated and dehydrogenated. Benzene, toluene, and mesitylene were identified in the appropriate fractions, which proved the presence of *cyclo*hexane, methyl*cyclo*hexane, and 1 : 3 : 5-trimethyl*cyclo*hexane in the original material. The formation of the last must be due to drastic degradation, followed by re-synthesis. The presence of a methylhydrindane in the material boiling between 165° and 185° is probable, but no decisive evidence was obtained.

EXPERIMENTAL.

Isomerisation of Decalin.—Commercial decalin was dried and distilled over sodium. The product used had b. p. 185—193°, $d_{4^\circ}^{20^\circ}$ 0·8781, $n_D^{20^\circ}$ 1·4771 and hence contained about 75% of the *trans*-isomeride. Hydriodic acid at 210° for periods up to 3 days had little effect on decalin. Aluminium chloride and bromide at 100° gave some low-boiling hydrocarbons, but the reaction was very slow. Temperatures of 130—140° were the optimum, as there was a tendency for charring at 150° or higher.

A mixture of 250 g. of decalin and 80 g. of aluminium chloride was heated for 24 hours under reflux in a flask fitted with a drying tube, the temperature being maintained at 130° by means of an electrically heated oven. The hydrocarbon was decanted from the viscous lower layer of aluminium chloride complex, washed with water and alkali, dried over calcium chloride, and fractionated through a jacketed 27 cm. column of Lessing rings at the rate of 1 drop/7—8 seconds. The yield and the physical properties of the various fractions obtained by one fractionation of the product from 1 kg. of decalin, were :

Boiling range	below 150°	150—155°	155—160°	160—165°	165—170°	170—175°	175—180°
Yield, g.	15	16	31	40	45	55	31 (Total, 23%)
$d_{4^\circ}^{17^\circ}$	—	0·822	0·829	0·837	0·840	0·852	0·861
$n_D^{17^\circ}$	—	1·435	1·448	1·453	1·456	1·461	1·464

From 50—70% of decalin was recovered; this was mainly the *trans*-form and boiled at 185—186°. Further fractionation revealed a peak at about 160° and in subsequent experiments the crude product was first roughly fractionated to remove the product boiling below 180°, and this was then slowly fractionated three times. The material examined in the subsequent work, which will be referred to as fraction A, had b. p. 158—163°, $d_{4^\circ}^{20^\circ}$ 0·8320, $n_D^{20^\circ}$ 1·4482 (yield, 2—5%). In all, 8 kg. of decalin were isomerised.

In the dehydrogenation experiments the activity of nickel–alumina catalysts was found to vary considerably with the method of preparation. Zelinski's method (*Ber.*, 1924, **57**, 667) was modified as follows : Concentrated sodium hydroxide solution was added to a solution of

240 g. of nickel nitrate ($6H_2O$) and 146 g. of aluminium nitrate ($9H_2O$) until this was alkaline; a rapid stream of carbon dioxide was then passed and the precipitate was collected, and dried at 120° without washing. The dried precipitate was boiled with water, collected, and dried with alcohol and ether. The light green powder was shaken with chopped asbestos, and the mixture introduced into the silica tube of the dehydrogenator. The catalyst was reduced in purified hydrogen at 300—330°. Decalin readily yielded naphthalene when passed over this catalyst.

Fraction A (15 g.) yielded only 330 c.c. of hydrogen when circulated over this catalyst until no further gas was evolved. This corresponds to about 3% of dehydrogenation. The product was extracted thrice with liquid sulphur dioxide, in which it was almost insoluble (unlike aromatic hydrocarbons). The undissolved hydrocarbon was washed with alkali and distilled over sodium. The product had b. p. 158—163°, $d_{4^\circ}^{20^\circ}$ 0·8323, $n_D^{20^\circ}$ 1·4483. It was insoluble in concentrated sulphuric acid, decolorised bromine very slowly and permanganate not at all (Found : C, 86·9; H, 13·0. $C_{10}H_{18}$ requires C, 87·0; H, 13·0%). As the physical properties were almost unaltered by this treatment, the experiments which follow were performed on fraction A without dehydrogenation.

A rapid stream of moist chlorine was passed through an ice-cooled U-tube containing fraction A and a crystal of iodine. After 5 hours the theoretical quantity for monochlorination had been absorbed; the product was dissolved in ether, washed with water and alkali, dried, and distilled. The main products were (i) recovered hydrocarbon, b. p. about 55°/15 mm., 23%; (ii) b. p. 80—90°/15 mm., 22%; (iii) b. p. 120—135°/15 mm., 5%; (iv) b. p. 135—170°/15 mm., 35%. Fractions (ii), (iii), and (iv) gave chlorine analyses corresponding approximately to those required for mono-, di- and tri-chloro-compounds. Fraction (ii) was stable in the air, but the higher fractions rapidly darkened. All had camphoraceous odours. Refractionation of (ii) gave the *monochloro*-compound (? II) in 20% yield; b. p. 88—91°/16 mm., $d_{4^\circ}^{20^\circ}$ 0·9823, $n_D^{20^\circ}$ 1·4753, $[R_L]_D$ 49·2 (calc., 48·8) (Found : Cl, 20·55. $C_{10}H_{17}Cl$ requires Cl, 20·6%). The monochloro-compound was unattacked by moist silver oxide in ether. Treatment with silver *p*-nitrobenzoate yielded *p*-nitrobenzoic acid and the olefin described below. This suggests that the chlorine is tertiary.

9 G. of the monochloro-compound were boiled with 9 g. of aniline for 2 hours; charring occurred. The product was cooled in ice and the mixture of hydrocarbon and aniline was decanted from the aniline hydrochloride and distilled. The unsaturated hydrocarbon (? III), b. p. 54—60°/20 mm., was washed with dilute acid and dried over calcium chloride. Yield, 30%. It had an unpleasant odour, decolorised permanganate and bromine, and was moderately stable in the air. It had $d_{4^\circ}^{20^\circ}$ 0·8632, n_D^{20} 1·4663, $[R_L]_D$ 43·6 (calc. $C_{10}H_{16}|\overline{\overline{1}}$ 43·6) (Found : C, 88·0; H, 11·9. $C_{10}H_{16}$ requires C, 88·2; H, 11·8%). Quinoline, dimethylaniline, and pyridine appeared to be without action on the chloro-compound.

To 14 g. of the unsaturated hydrocarbon mechanically stirred in aqueous sodium carbonate, 2% potassium permanganate was added slowly during 8 hours until no more was absorbed. The solution was filtered from manganese mud, cleared with sulphur dioxide, acidified, and extracted with ether for 24 hours. The residue from the extract was purified with sodium bicarbonate solution in the usual way, 3·5 g. of an acid gum being obtained. This gave a dense precipitate of iodoform (m. p. and mixed m. p.) with alkali and iodine in potassium iodide. After several weeks it deposited a small quantity of diamond-shaped crystals, m. p. 120° after recrystallisation from water. This acid (Found : C, 64·9; H, 8·7. $C_{10}H_{16}O_3$ requires C, 65·2; H, 8·7%) (? *3-acetyl-1-methyl*cyclo*pentane-2-acetic acid*, IV) yielded iodoform and gave a semicarbazone, m. p. 195° (from acetic acid). No other solid degradation products could be isolated.

Lower-boiling fractions. The material boiling below 150°, formed by the action at 130° of aluminium chloride on 7 kg. of decalin, yielded the following fractions :

Boiling range...	to 76°	76—80°	80—90°	90—100°	100—110°	110—120°	120—130°	130—140°
$d_{4^\circ}^{20^\circ}$	0·737	0·757	0·761	0·766	0·771	0·768	0·770	0·781
$n_D^{20^\circ}$	1·404	1·414	1·416	1·418	1·422	1·420	1·421	1·426
Yield, g..........	14	20	17	32	17	43	21	33

These fractions were dehydrogenated by the method already described and the products were separated into aromatic and hydroaromatic portions by means of liquid sulphur dioxide. The products were redistilled, there being no substantial change in b. p. The following fractions *soluble* in sulphur dioxide were isolated :

Boiling range ...	80—90° (*a*)	90—100°	100—110° (*b*)	110—120°	120—130°	130—140° (*c*)
$d_{4^\circ}^{20^\circ}$	0·873	—	0·796	—	0·798	0·830
$n_D^{20^\circ}$	1·500	1·476	1·441	1·423	1·446	1·451

The fractions *insoluble* in sulphur dioxide were very similar to the corresponding undehydrogenated material in physical properties. Fraction (*a*) was almost pure benzene from its physical properties and analysis (Found : C, 91·5; H, 8·0. Calc. : C, 92·3; H, 7·7%). It solidified in a freezing mixture. The physical properties of fraction (*b*) showed that dehydrogenation was incomplete, but nitration yielded 2 : 4-dinitrotoluene (m. p. and mixed m. p. 70°). Nitration of fraction (*c*) yielded 2 : 4 : 6-trinitromesitylene in poor yield (m. p. and mixed m. p. 231—232°). No other solid derivatives could be isolated from the dehydrogenated fractions.

Syntheses.—(a) *Attempted preparation of* 1 : 4-*dimethyl*bicyclo*octane* (I). α-Methyladipic ester (Dieckmann, *Annalen*, 1901, **317**, 69) was cyclised to ethyl 5-methyl*cyclo*pentanone-2-carboxylate in 70—80% yield, following the details used by Linstead and Meade (*loc. cit.*) in the case of adipic ester. The sodio-compound from 250 g. of the keto-ester, prepared in dry benzene (1 kg.) by the use of " molecular " sodium (34 g.), was treated with a slight excess of ethyl α-bromopropionate (270 g.), and the reaction completed by refluxing for 8 hours. The product, isolated in the usual way, was fractionated into *ethyl* 5-*methyl*cyclo*pentanone*-2-*carboxylate*-2-α-*propionate*, b. p. 171—173°/19 mm. (65%), and the corresponding open-chain ester, b. p. 180—190°/20 mm. The cyclic keto-ester had a rather garlic-like odour, gave no colour with ferric chloride, and, like other substances of similar structure, failed to condense under the usual conditions with salts of phenylhydrazine or semicarbazide (Found : C, 61·7; H, 8·1. $C_{14}H_{22}O_5$ requires C, 62·2; H, 8·1%). 100 G. were refluxed with 600 c.c. of concentrated hydrochloric acid for 24 hours, and the reagent then removed under reduced pressure. The residue on fractionation yielded 75% of 5-*methyl*cyclo*pentanone*-2-α-*propionic acid*, b. p. 152°/6—7 mm., as a gum which solidified to small diamond-shaped crystals, m. p. 76° (from light petroleum) (Found : C, 63·3; H, 8·4. $C_9H_{14}O_3$ requires C, 63·5; H, 8·2%). The colourless phenylhydrazone melted at 192°. 70 G. of the acid were refluxed for 8 hours with 100 c.c. of ethyl alcohol, 150 c.c. of benzene, and 20 c.c. of concentrated sulphuric acid. The product was washed with water and sodium bicarbonate solution, and dried; the residue after removal of the benzene yielded 95% of the *ethyl* ester, b. p. 127—128°/12 mm., $d_{4^\circ}^{24^\circ}$ 1·012, $n_D^{24^\circ}$ 1·4468 (Found : C, 66·4; H, 9·0. $C_{11}H_{18}O_3$ requires C, 66·6; H, 9·1%). This substance failed to condense with cyanoacetic ester in the presence of piperidine at atmospheric pressure (compare Barrett, Cook, and Linstead, J., 1935, 1065) and condensation by means of potassium ethoxide (Kon and Nanji, J., 1932, 2429) gave no useful product.

(b) *Preparation of* 2 : 5-*dimethyl*bicyclo*octane* (V). The crude β-methyladipic acid obtained by nitric acid oxidation of 500 g. of 4-methyl*cyclo*hexanol was refluxed on the steam-bath for 8 hours with a mixture of 650 c.c. of absolute alcohol, 125 c.c. of concentrated sulphuric acid, and 1½ l. of benzene. The benzene layer yielded 500 g. of β-methyladipic ester, b. p. 138—144°/16—17 mm. This was cyclised with 1½ mols. of sodium in 2½ l. of benzene in the usual way and yielded 220 g. of ethyl 4-methyl*cyclo*pentanone-2-carboxylate, b. p. 123—125°/24 mm. (compare Dieckmann, *Annalen*, 1901, **317**, 78). Overall yield, 30% from methyl*cyclo*hexanol.

200 G. of this keto-ester were added slowly to 27 g. of " molecular " sodium in 1·5 l. of benzene. The sodio-compound formed slowly and reaction was completed by 5 hours' refluxing. 145 G. of chloroacetic ester were then added, and the mixture refluxed over-night and worked up in the usual manner, unchanged material being removed with 10% caustic potash solution. *Ethyl* 4-*methyl*cyclo*pentanone*-2-*carboxylate*-2-*acetate*, b. p. 180—183°/24 mm., was isolated in 65% yield. It gave no colour with ferric chloride and no semicarbazone (Found : C, 60·5; H, 7·5. $C_{13}H_{20}O_5$ requires C, 60·9; H, 7·8%). Some open-chain material was also formed.

100 G. of this ester were refluxed for 6 hours with 500 c.c. of concentrated hydrochloric acid, the mineral acid removed under reduced pressure, and the product distilled. The crude 4-methyl*cyclo*pentanone-2-acetic acid (b. p. 183—190°/24 mm.; yield, 76%) obtained was esterified after one distillation. A solution of 90 g. in 150 c.c. of ethyl alcohol was saturated with hydrogen chloride for ½ hour, kept for 4 hours, and worked up in the usual way. The neutral fraction yielded 68% of *ethyl* 4-*methyl*cyclo*pentanone*-2-*acetate*, b. p. 130—133°/23 mm., $d_{4^\circ}^{21\cdot5^\circ}$ 1·0217, $n_D^{21\cdot5^\circ}$ 1·4439, $[R_L]_D$ 47·2 (calc., 47·6) (Found : C, 65·5; H, 8·3. $C_{10}H_{16}O_3$ requires C, 65·2; H, 8·7%). The semicarbazone melted at 141°.

220 C.c. of calcium-dried alcohol were treated successively with 17 g. of potassium and 49 g. of cyanoacetic ester; the whole was cooled in a freezing mixture and treated with 80 g. of the 4-methyl*cyclo*pentanoneacetic ester (compare Kon and Nanji, *loc. cit.*). The product was kept at 0° for 1 day and at room temperature for 2 days, treated with ice and dilute acid, and extracted with ether. The extract was shaken with sodium bicarbonate solution and water, and dried. The residue from the ether yielded 30 g. of *ethyl* 4-*methyl*cyclo*pentylidene*-1-*cyanoacetate*-2-*acetate*, b. p. 194°/4 mm., and a further quantity of this was obtained by esterifying

the acid fraction. Total yield, 35%; $d_{4^\circ}^{21^\circ}$ 1·0738, $n_D^{21^\circ}$ 1·4782 (Found: C, 65·0; H, 7·3. $C_{15}H_{21}O_4N$ requires C, 64·6; H, 7·5%). 42 G. of the ester in 100 c.c. of alcohol were reduced over 1½ g. of Adams's platinum catalyst in 24 hours. *Ethyl 4-methyl*cyclo*pentane-1-cyanoacetate-2-acetate* (92% yield) had b. p. 205°/17 mm., $d_{4^\circ}^{22^\circ}$ 1·0535, $n_D^{22^\circ}$ 1·4584, $[R_L]_D$ 72·8 (calc., 72·3) (Found: C, 63·8; H, 8·2. $C_{15}H_{23}O_4N$ requires C, 64·1; H, 8·2%).

The cyano-diester (42 g.) was refluxed with hydrochloric acid for 10 hours, and the product distilled in steam. The distillate gave a little of the semicarbazone of 5-methyl*bicyclo*octanone (compare Linstead and Meade, *loc. cit.*). The residue deposited *4-methyl*cyclo*pentane-1 : 2-diacetic acid* (38%) as crystalline flakes, very soluble in hot water, almost insoluble in cold, m. p. 173—174° (Found: C, 60·2; H, 7·95. $C_{10}H_{16}O_4$ requires C, 60·0; H, 8·0%). 9 G. of the diacetic acid were heated to 270—280° with 0·9 g. of baryta. Yield of 5-methyl-0 : 3 : 3-*bicyclo*octan-2-one (VI), 2 g. (32%). The *semicarbazone*, recrystallised from rectified spirit, had m. p. 183° (Found: C, 61·3; H, 8·8; N, 21·7. $C_{10}H_{17}ON_3$ requires C, 61·6; H, 8·7; N, 21·5%). From this the ketone was regenerated, b. p. 104°/20 mm., $d_{4^\circ}^{24^\circ}$ 0·9666, $n_D^{24^\circ}$ 1·4544, $[R_L]_D$ 39·3 (calc., 39·39).

The Grignard compound prepared from 0·4 g. of magnesium and 3 g. of methyl iodide in 20 c.c. of dry ether was treated with 1·6 g. of the ketone. After an hour, the mixture was cooled to 0° and decomposed with ice-cold dilute sulphuric acid, and the product isolated in the usual way. The alcohol was dehydrated, without purification, by slow distillation with 5% (0·05 g.) of aniline hydrobromide. *2 : 5-Dimethyl-*Δ^1*-0 : 3 : 3-*bicyclo*octene* (VII) distilled at 167°; it was kept for a day over water, dried, and distilled. It had a fish-like smell and reduced permanganate. Yield, 1·0 g. (70%); $d_{4^\circ}^{24^\circ}$ 0·8605, $n_D^{24^\circ}$ 1·4649, $[R_L]_D$ 43·7 (calc. for $C_{10}H_{16}|\overline{1}$, 43·51) (Found: C, 88·0; H, 11·5. $C_{10}H_{16}$ requires C, 88·2; H, 11·8%).

This hydrocarbon (0·8 g.) was hydrogenated over Adams's platinum catalyst (0·005 g.) without a solvent. The theoretical quantity of hydrogen was absorbed in 6 hours. The *2 : 5-dimethyl-0 : 3 : 3-*bicyclo*octane* (V) after distillation did not attack permanganate and had a faint odour resembling that of the isomerisation product of decalin. Yield, 0·75 g.; b. p. 165—167°/760 mm., $d_{4^\circ}^{24^\circ}$ 0·8341, $n_D^{24^\circ}$ 1·4481, $[R_L]_D$ 44·25 (calc., 43·98) (Found: C, 86·8; H, 13·0. $C_{10}H_{18}$ requires C, 87·0; H, 13·0%).

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