#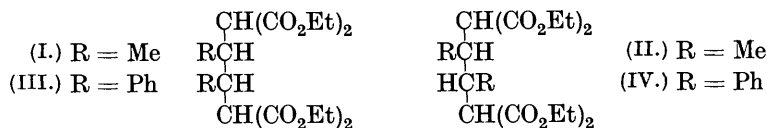 CXXXIV.—*Synthesis of Cyclic Compounds. Part II. Ethyl* r- *and* meso-βγ-*Diphenylbutane-ααδδ-tetra-carboxylates. Synthesis of a Truxinic Acid.*

By Israel Vogel.

In Part I (J., 1927, 1986) the synthesis of ethyl βγ-dimethylbutane-ααδδ-tetracarboxylate, a substance which should theoretically exist in a *meso*- (I) and a racemic form (II), was described. The separation

		$CH(CO_2Et)_2$	$CH(CO_2Et)_2$	
(I.) R = Me		RCH	RCH	(II.) R = Me
(III.) R = Ph		RCH	HCR	(IV.) R = Ph
		$CH(CO_2Et)_2$	$CH(CO_2Et)_2$	

of the two esters (I) and (II) could not be effected owing to the proximity of their boiling points; hydrolysis by alcoholic potash, however, yielded a mixture of the two corresponding stereoisomeric tetracarboxylic acids which was separated into two forms, m. p. 152—154° (decomp.) and 185—185·5° (decomp.), respectively. The *dl*-configuration then provisionally assigned to the less fusible form has now been confirmed by the resolution of the acid ·into optically active components *via* the strychnine salts, full details of which will be given in a later communication.
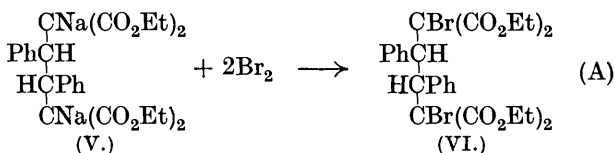
Treatment of the mixture of esters (I) and (II) with sodium methoxide and bromine gave an almost quantitative yield of the corresponding *cyclo*butane compound (compare Perkin, J., 1887, **51**, 1; 1894, **65**, 572), in agreement with the requirements of the Thorpe–Ingold modified strain theory.

The investigation of the analogous βγ-diphenyl compounds was obviously desirable, since it would not only provide a means of studying the effect of two adjacent $\frac{Ph}{H}{>}C{<}^ε_θ$ groups on this method of *cyclo*butane ring formation, but would also lead to a rational synthesis of the truxinic acids. Unfortunately, no calcul-ations of the value of the angle θ are at present available, so that no predictions of the ease of ring formation can be made on purely spatial grounds. It would appear, however, that in addition to the " volume " factor, there is a " polar " factor associated with the phenyl group which must be taken into account.
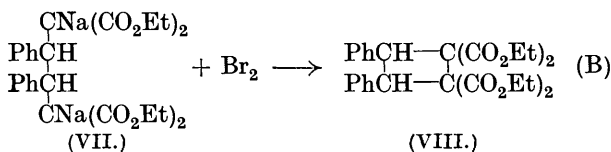
Henle (*Annalen*, 1906, **348**, 16), on reducing ethyl benzylidene-malonate with aluminium amalgam, was able to isolate only ethyl benzylmalonate. Reduction under the conditions previously described (Vogel, J., 1927, 594) gave, however, in addition to 50—55% of ethyl benzylmalonate, 35—40% of a mixture of two *ethyl*

L L 2

βγ-diphenylbutane-ααδδ-tetracarboxylates.   This mixture was quantitatively separated by means of absolute alcohol (see p. 1020) and shown to consist of about 40% of a solid form, m. p. 88°, to which the racemic configuration (IV) is assigned, and about 60% of a liquid *meso*-form (III).   Hydrolysis with alcoholic potash gave the two corresponding tetracarboxylic acids, m. p. 219—220° (decomp.) and 182—183° (decomp.), respectively.

Both esters form disodium compounds, the liquid more readily than the solid form, on treatment with a suspension of sodium methoxide in dry ether.   Attempts to produce ring closure by means of bromine led to widely different results in the two cases. The *disodium* compound (V) from the solid modification reacted with twice the calculated quantity of bromine required for *cyclo*butane ring formation and gave a nearly quantitative yield of *ethyl* r-αδ-*dibromo-βγ-diphenylbutane-ααδδ-tetracarboxylate* (VI),   a crystalline solid, m. p. 180°, the reaction evidently proceeding according to scheme (A).   This reaction together with the fact that the solid yields the higher-melting tetracarboxylic acid may be taken as establishing the *dl*-configuration of the ester, m. p. 88°.
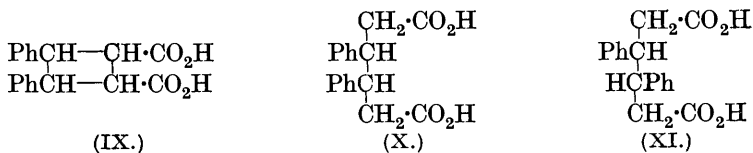
$$
\begin{array}{ccc}
\underset{(V.)}{\overset{\displaystyle \begin{array}{l} \text{CNa(CO}_2\text{Et)}_2 \\ \text{PhCH} \\ \text{HCPh} \\ \text{CNa(CO}_2\text{Et)}_2 \end{array}}{}} & + \; 2\text{Br}_2 \;\longrightarrow\; & \underset{(VI.)}{\overset{\displaystyle \begin{array}{l} \text{CBr(CO}_2\text{Et)}_2 \\ \text{PhCH} \\ \text{HCPh} \\ \text{CBr(CO}_2\text{Et)}_2 \end{array}}{}} \quad (A)
\end{array}
$$

The *disodium* compound (VII) derived from the liquid form, on the other hand, reacted with the calculated quantity of bromine required for *cyclo*butane ring formation (B), presumably with the formation

$$
\begin{array}{ccc}
\underset{(VII.)}{\overset{\displaystyle \begin{array}{l} \text{CNa(CO}_2\text{Et)}_2 \\ \text{PhCH} \\ \text{PhCH} \\ \text{CNa(CO}_2\text{Et)}_2 \end{array}}{}} & + \; \text{Br}_2 \;\longrightarrow\; & \underset{(VIII.)}{\overset{\displaystyle \begin{array}{l} \text{PhCH—C(CO}_2\text{Et)}_2 \\ \text{PhCH—C(CO}_2\text{Et)}_2 \end{array}}{}} \quad (B)
\end{array}
$$

of the ester (VIII).   Hydrolysis of the product with alcoholic potash, which was accompanied by considerable carbonisation and the formation of resinous matter, and subsequent heating at 280° until effervescence ceased gave an extremely poor yield (less than 1%) of an acid, m. p. 239°, which was analysed in the form of its dimethyl ester, m. p. 117°.   There is no doubt that this is the *cyclo*butane acid (IX), since the two βγ-diphenyladipic acids, which should theoretically possess the configurations (X) and (XI), melt respectively at 169—170° and 270°, the dimethyl esters melting at 73° and 175° (Henle, *loc. cit.*).   These results were confirmed by

treating the mixed disodio-compounds derived from the mixture of esters (III) and (IV) with bromine : the quantity of bromine

$$
\begin{array}{ccc}
 & CH_2 \cdot CO_2H & CH_2 \cdot CO_2H \\
PhCH-CH \cdot CO_2H & PhCH & PhCH \\
PhCH-CH \cdot CO_2H & PhCH & HCPh \\
 & CH_2 \cdot CO_2H & CH_2 \cdot CO_2H \\
\text{(IX.)} & \text{(X.)} & \text{(XI.)}
\end{array}
$$

required was equal to that calculated on the assumption that 45% of the disodio-compound had reacted according to scheme (A) and the remainder according to (B). The dibromo-ester (VI), which is only slightly soluble in both ether and water, was precipitated on the addition of water, the product of reaction (A), which was identical with that obtained from the *meso*-form, remaining in the ethereal solution.

The greater rigidity of structure associated with a molecule containing a phenyl group is clearly shown by these experiments, since the mixture of ethyl *r*- and *meso*-dimethylbutanetetra-carboxylates gives an excellent yield of the *cyclo*butane compound, thus showing that inversion must have occurred during ring form-ation. This rotation is apparently inhibited for the diphenyl com-pounds and the *r*-dibromo-ester (VI) is produced.

There are six possible configurations for the 2 : 3-*diphenyl*cyclo-*butane*-1 : 4-*dicarboxylic acid* (IX), or truxinic acid if the nomen-clature of Stoermer (*Ber.*, 1922, **55**, 1868) be adopted. Of these, only four, the β-, δ-, ζ-, and *neo*-acids, have so far been isolated from cinnamic acid by additive ring formation (compare *Ann. Rep.*, 1924, **21**, 96). The melting points of these acids and their dimethyl esters are respectively 209° (decomp.) and 76°, 175° and 77°, 239° and 116°, 209° and 127° (Stoermer and Bachér, *Ber.*, 1922, **55**, 1865). The properties of the synthetical acid and its dimethyl ester are in complete agreement with those which have been described for ζ-truxinic acid and it would appear that they are identical; a direct comparison, however, has not yet been possible.

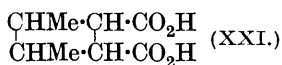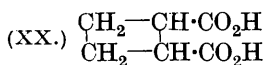The ζ- and δ- and also the β- and *neo*-acids are related as *cis*- and *trans*-acids.

These two pairs of acids are closely allied to the *cis*- and *trans*-2 : 3-dimethyl*cyclo*butane-1 : 4-dicarboxylic acids described in Part I and it appears reasonable to assign, at least provisionally, con-figurations to the synthetical acids similar to those deduced for the truxinic acids from the work of Stobbe, Stoermer, de Jong, and their co-workers. The configuration (XV) is assigned to the author's synthetical diphenyl acid and it appears very probable that *cis*- and *trans*-2 : 3-dimethyl*cyclo*butanedicarboxylic acids have configurations

similar to (XV) and (XVII).   The constitution, analogous to (XIII), originally assigned to the *cis*-acid is now considered less likely than (XV).
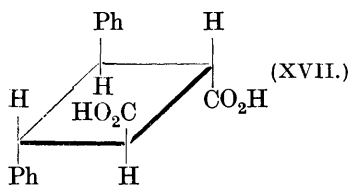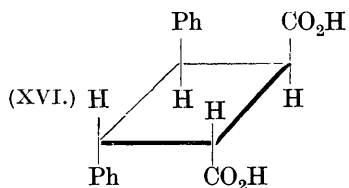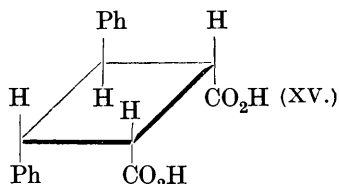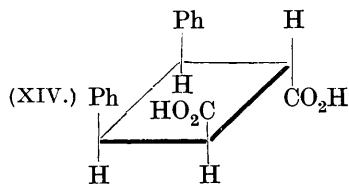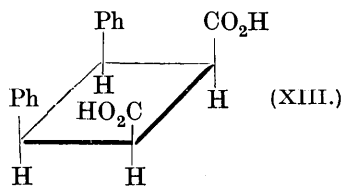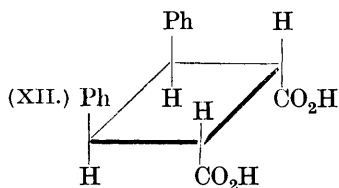
The very small yield of *cyclo*butane compounds obtained from the ethyl βγ-diphenyl- in contrast with the excellent yields obtained from the unsubstituted (Perkin, *loc. cit.*) and βγ-dimethyl-butanetetra-carboxylates (J., 1927, 1985) illustrates the complexity of the problem of explaining the effect of substituents on the *ease of ring formation.*   Although this is doubtless due to stereochemical influences such as are postulated by the modified strain theory, the possibility that a polarity factor may play some part must not be overlooked.   No experiments have yet been completed on the effect of substituent groups on the *stability* of *cyclo*butane rings, but the results already recorded in the literature are in complete accord with the Thorpe–Ingold theory; for example, Perkin and Simonsen (J., 1909, **95**, 1164) found that whereas fission of the *cyclo*butane ring

$$\text{CO}_2\text{H·CH}\underset{\text{CH}_2}{\overset{\text{CH}_2}{<}}\text{>CH·CO}_2\text{H} \qquad \text{CO}_2\text{H·CH}\underset{\text{CH}_2}{\overset{\text{CMe}_2}{<}}\text{>CH·CO}_2\text{H}$$
$$\text{(XVIII.)} \qquad\qquad\qquad\qquad \text{(XIX.)}$$

$$\text{(XX.)}\quad \begin{array}{l}\text{CH}_2\text{—CH·CO}_2\text{H}\\ \text{CH}_2\text{—CH·CO}_2\text{H}\end{array} \qquad \begin{array}{l}\text{CHMe·CH·CO}_2\text{H}\\ \text{CHMe·CH·CO}_2\text{H}\end{array}\ \text{(XXI.)}$$
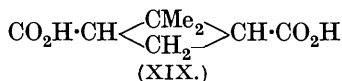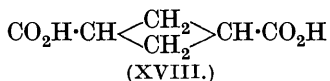
in *cis-cyclo*butane-1 : 3-dicarboxylic acid (XVIII) could readily be effected by heating with fuming hydrobromic acid in a sealed tube

at 100°, *cis*-norpinic acid (XIX) was attacked neither by this reagent at 120° nor by concentrated sulphuric acid at 100°. It should, however, be mentioned that the stability of the ring is affected by the *relative* positions of the substituent groups round the ring, since it has been shown (Perkin and Simonsen, *loc. cit.*) that the isomeric unsubstituted 1 : 4-dicarboxylic acid is unaffected by heating with fuming hydrobromic acid at 100°. However, in their stability towards different reagents, Perkin's unsubstituted 1 : 4-dicarboxylic acids (XX) may be compared with the author's dimethyl homologues (XXI), and it may be expected on the basis of the modified strain theory that the difference in stability will not be very great but that on the whole (XXI) will be slightly more stable than (XX). It is hoped to test this point in the near future.
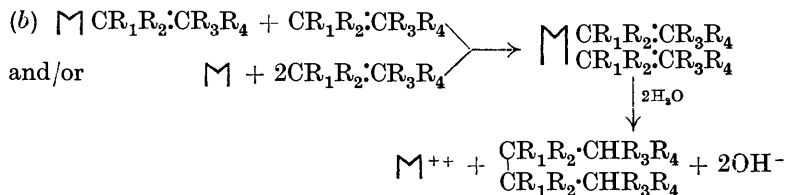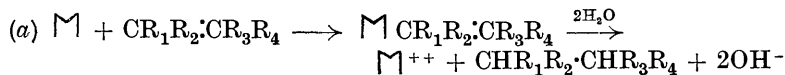
The substances being investigated in this series of researches are all conjugated compounds of the general formula $CR_1R_2{:}CR_3R_4$, where R_1, R_2 are groups or ring systems of very varied type and R_3, R_4 are H, CN, CO_2Et, COMe, etc. On reduction with moist aluminium amalgam in ether, two simultaneous and also possibly consecutive reactions take place, *viz.*, (*a*) the addition of hydrogen to the double bond with the production of compounds (XXII), and (*b*) the formation of bimolecular compounds (XXIII). Most of the

$$\text{(XXII.)} \quad CHR_1R_2{\cdot}CHR_3R_4 \qquad \begin{array}{l} CR_1R_2{\cdot}CHR_3R_4 \\ CR_1R_2{\cdot}CHR_3R_4 \end{array} \text{ (XXIII.)}$$

recorded cases (compare ethyl ethylidenemalonate, Higginbotham and Lapworth, J., 1923, **123**, 1618; Vogel, J., 1927, 1985 : mesityl oxide, Vogel, J., 1927, 594) have shown that the reaction proceeds almost exclusively in the direction (*b*). The example now described, *i.e.*, the reduction of ethyl benzylidenemalonate, shows clearly that in this instance reactions (*a*) and (*b*) both take place, 50 to 55% of (XXII) and 35—40% of (XXIII) being formed. The extent to which each of these reactions takes place is controlled mainly by the groups R_1, R_2; and it appears that the volume of these groups plays an important part, provided of course that R_3 and R_4 are suitable " activating " groups to enable the reduction to take place at all. It seems that the larger the volume of R_1, R_2 the more reaction (*a*) takes place in preference to (*b*). This view receives support from the fact that ethyl α-cyano*cyclo*hexylideneacetate gives about 80% of ethyl *cyclo*hexylcyanoacetate and 5—10% of a solid which is probably of type (XXIII). Full details of this and analogous cases will be given in a subsequent communication.

The mechanism of the reduction of unsaturated compounds in ethereal solution by moist aluminium amalgam adopted by the author is based on that originally suggested by Higginbotham and

Lapworth (J., 1923, **123**, 1618).   The unsaturated compound is first adsorbed on the surface of the metal and reaction takes place at the surface, where chemical forces are known to be very powerful.   Two modes of reaction corresponding to (a) and (b) are possible and may be represented as follows :

(a) $\text{M} + CR_1R_2{:}CR_3R_4 \longrightarrow \text{M } CR_1R_2{:}CR_3R_4 \xrightarrow{2H_2O}$
$$\text{M}^{++} + CHR_1R_2{\cdot}CHR_3R_4 + 2OH^-$$

(b) $\text{M } CR_1R_2{:}CR_3R_4 + CR_1R_2{:}CR_3R_4$

and/or $\text{M} + 2CR_1R_2{:}CR_3R_4$

$$\searrow \quad \text{M} \begin{matrix} CR_1R_2{:}CR_3R_4 \\ CR_1R_2{:}CR_3R_4 \end{matrix}$$

$$\downarrow 2H_2O$$

$$\text{M}^{++} + \begin{matrix} CR_1R_2{\cdot}CHR_3R_4 \\ CR_1R_2{\cdot}CHR_3R_4 \end{matrix} + 2OH^-$$

This " adsorption " theory receives support from the fact that the yields of reduction product under otherwise comparable conditions are controlled by the thickness, and therefore the surface, of the aluminium foil employed in the preparation of the amalgam. Further, there is always an initial period of induction and this is doubtless associated with the adsorption of the unsaturated compound.   The mechanism suggested by Cohen (*Rec. trav. chim.*, 1919, **38**, 113) involving the initial addition of a hydrogen atom to the carbonyl oxygen with the formation of a tervalent carbon derivative which subsequently polymerises with the formation of a bimolecular compound, and also his explanation of the production of the unimolecular reduction product by fission of the bimolecular compound, are considered improbable.   This mechanism has been adopted by Steinkopf and Wolfram (*Annalen*, 1923, **430**, 128) as a general explanation of the reduction of unsaturated compounds, although these authors assume that the unimolecular compound is produced by the reduction of the free radical.   The present author's view is that the mechanism of the reduction depends on the nature of the reducing agent and that while the " free radical " theory may or may not hold for other types of reduction (compare Conant and Cutter, *J. Amer. Chem. Soc.*, 1926, **48**, 1026, who found that mesityl oxide is not reduced by vanadous or chromous chloride, the operation of another mechanism thus being indicated), all the results of experiments on reduction with aluminium amalgam in ethereal solution are in complete agreement with the " adsorption " theory outlined above.

EXPERIMENTAL.

Ethyl benzylidenemalonate was prepared by condensing freshly distilled benzaldehyde with ethyl malonate in the   presence   of

piperidine (Knoevenagel, *Ber.*, 1898, **31**, 2585). The pure substance boiled at 180°/14 mm. and had $d_4^{18\cdot4^\circ}$ 1·1048 and $n_D^{18\cdot4^\circ}$ 1·53795 (supercooled liquid).

Reduction with Moist Aluminium Amalgam. Preparation of Ethyl meso- *and* r-βγ-*Diphenylbutane-*ααδδ-*tetracarboxylates* (III and IV).—Ethyl benzylidenemalonate (150 g.) was reduced by moist aluminium amalgam (200 g.) (Vogel, *J.*, 1927, 594). The product, after being worked up in the usual manner, was fractionated under diminished pressure (water-pump); 75—80 g. were collected up to 205°/20 mm. (mainly at 174—178°/17 mm.) and the pressure then began to fall. The residue (60—65 g.), a dark red, extremely viscid mixture of the *meso*- and *r*-esters (III) and (IV) which partly crystallised on standing, was distilled (oil-pump) as rapidly as possible, in order to reduce carbonisation to a minimum. A very small quantity distilled at 245—255°/3 mm. The main fraction, b. p. 255—270°/3 mm., an almost colourless, viscid liquid which darkened on exposure to the atmosphere, partly crystallised when left over caustic potash in a vacuum desiccator for 24 hours. After the adhering oil had been removed by draining on tile and trituration with absolute alcohol, *ethyl* r-βγ-*diphenylbutane-*ααδδ-*tetracarboxylate* was obtained in beautiful crystals, m. p. 88° (Found : C, 67·3; H, 6·8. $C_{28}H_{34}O_8$ requires C, 67·5; H, 6·9%).

The *meso*-ester was isolated as a viscid red oil by extracting the porous tiles with ether and was analysed in the form of the corresponding tetracarboxylic acid (*vide infra*).

The fraction of low b. p. obtained from the reduction product, on redistillation, gave ethyl benzylmalonate, b. p. 163°/14 mm., $d_4^{20\cdot0^\circ}$ 1·0750, $n_D^{20\cdot0^\circ}$ 1·4872 (Found : C, 67·3; H, 7·2. Calc.: C, 67·2; H, 7·2%), which was hydrolysed by alcoholic potash to benzylmalonic acid; this, after crystallising from benzene, melted, alone or mixed with an authentic specimen prepared from benzyl chloride and ethyl sodiomalonate, at 120° (decomp.). Henle (*loc. cit.*) gives m. p. 114—115°.

Hydrolysis of the r-*Ester* (IV).—A mixture of 5 g. of the solid ester, 10 g. of absolute alcohol, and 4 g. of caustic potash in 8 g. of water was refluxed for 5 hours and then evaporated to dryness. A solution of the residue in a little water was extracted with ether to remove unchanged ester, if present, and acidified with dilute sulphuric acid, and the precipitated *r-acid* was washed with water and freed from potassium sulphate by dissolution in warm acetone and from a small quantity of the *meso*-acid by washing with dry ether; it then melted at 219—220° (decomp.) (Found : C, 62·5; H, 4·9; equiv. by titration, 98. $C_{20}H_{18}O_8$ requires C, 62·2; H, 4·7%; equiv., 96·5).

Preparation of the meso-*Acid.*—The thick oil obtained by ether extraction of the porous tiles in the separation of the *r*-ester was hydrolysed exactly as described for the *r*-ester. The meso-*acid* had m. p. 182—183° (slight softening at 172°) (Found : C, 62·2; H, 4·8%; equiv. by titration, 97).

Quantitative Separation of the meso- *and* r-*Esters* (III and IV).— The redistilled ethyl diphenylbutanetetracarboxylate mixture (32 g.) was agitated with about an equal weight of absolute alcohol. The *r*-ester, which rapidly crystallised after several hours, was collected and washed with absolute alcohol; further crops were obtained by concentrating the filtrate at room temperature under reduced pressure until no more crystals would separate. The final liquor consisted of practically pure *meso*-ester. In this way 13 g. of the *r*-ester and 19 g. of the *meso*-ester (a red viscid oil, weighed after being kept in a vacuum over concentrated sulphuric acid for a week) were obtained.

Reaction of the Disodio-compound (V) *of Ethyl* r-βγ-*Diphenylbutane-*ααδδ-*tetracarboxylate with Bromine. Formation of Ethyl* r-αδ-*Di-bromo-*βγ-*diphenylbutane-*ααδδ-*tetracarboxylate* (VI).—To 0·80 g. of " molecular " sodium under 100 c.c. of dry ether, 1·45 c.c. of dry methyl alcohol were added. When all the sodium had reacted (gentle warming was necessary), a solution of the *r*-ester (IV) (8·3 g.) in dry ether (175 c.c.) was added. The *disodio*-compound (V) gradually separated and after 12 hours it was filtered off, washed rapidly with absolute ether containing a little absolute alcohol, and dried over concentrated sulphuric acid in a vacuum (Found : Na, 8·7. $C_{28}H_{32}O_8Na_2$ requires Na, 8·5%).

In another experiment in which the same quantities were employed, the suspension of the disodio-compound in ether was stirred mechanically, and dry bromine (2·0 c.c.) slowly added; a yellow colour indicative of the presence of excess of bromine, which could also be identified by its odour, became apparent after 1·90 c.c. had been added (calc. according to reaction A, p. 1014, 1·88 c.c.). The stirring was continued for 3 hours, water was then added, the resultant solution filtered, and the residue (4·0 g., m. p. 174°) washed with water and with ether; it then crystallised from ethyl acetate in colourless, glistening plates, m. p. 180° (Found : C, 51·0; H, 4·9; Br, 24·7. $C_{28}H_{32}O_8Br_2$ requires C, 51·2; H, 4·9; Br, 24·4%). The ethereal solution was washed with dilute sodium carbonate solution and with water, dried with anhydrous sodium sulphate, and evaporated. The pale red, viscid residue (7 g.) crystallised almost completely when left in contact with absolute alcohol over caustic potash in a vacuum desiccator; after being washed with alcohol and recrystallised from ethyl acetate, it melted at 180° and was

identical with the precipitated solid. No other reaction product could be isolated.

Reaction of the Disodio-compound (VII) *of Ethyl* meso-$\beta\gamma$-*Diphenyl-butane-$\alpha\alpha\delta\delta$-tetracarboxylate with Bromine. Synthesis of a* 2 : 3-*Di-phenyl*cyclo*butane*-1 : 4-*dicarboxylic Acid* (*Truxinic Acid*) (IX).— The *disodio*-compound (VII) was prepared, exactly as described for the *r*-compound, as a pale yellow solid (Found : Na, 8·6%). The quantities employed in a particular experiment were 1·25 g. of " molecular " sodium, 2·25 c.c. of dry methyl alcohol, and 12· 7 g. of ester in 100 c.c. of absolute ether. The suspension of the disodio-compound in dry ether was mechanically stirred and slowly treated with dry bromine : excess of bromine was apparent when 1·40 c.c. had been added (calc. for *cyclo*butane ring formation according to reaction B, p. 1014, 1·39 c.c.). Water was added after 3 hours' stirring, and no solid remained undissolved (compare *r*-compound). After the product had been worked up in the usual manner, 12 g. of a viscid, dark red oil remained. This was hydrolysed by heating on the steam-bath for 18 hours with twice the calculated quantity of ethyl-alcoholic potash, and the solution evaporated to dryness. A solution of the residue in water was filtered to remove carbonaceous matter and then cautiously acidified with ice-cold dilute sulphuric acid. The precipitated resinous matter was removed by filtration and the filtrate was saturated with ammonium sulphate and extracted seven times with ether. The ethereal solution, dried with anhydrous sodium sulphate and evaporated, yielded a pale yellow, oily solid which crystallised almost completely when left in contact with light petroleum (b. p. 40—60°). The slightly gummy, hygroscopic solid was heated at 280° until effervescence ceased, and the residue was fractionally crystallised from acetone ; it was thus possible to isolate a solid, which melted at 239°. It was found best, however, to esterify the product with methyl alcohol and sulphuric acid and treat the brown gummy esterification product with large quantities of light petroleum (b. p. 40—60°) ; on allowing the latter to evaporate, a somewhat oily crystalline solid was obtained. This was drained on tile and recrystallised from dilute methyl alcohol ; it then melted at 117°. By repeating this process with the resinous matter, which gradually solidified, obtained on acidifying the alkaline solution, further small quantities of the ester, m. p. 117°, could be obtained. The total yield of pure ester in one experiment rarely exceeded a few mg. (Found : C, 74·0 ; H, 6·2. $C_{20}H_{20}O_4$ requires C, 74·1 ; H, 6·2%).

Reaction of Bromine with the Disodio-compounds of the Mixture of Ethyl meso- *and* r-$\beta\gamma$-*Diphenylbutane-$\alpha\alpha\delta\delta$-tetracarboxylates.*—The mixture of the esters was converted into the disodio-compounds

(2·25 g. of " molecular " sodium, 100 c.c. of ether, 4·00 c.c. of methyl alcohol, 22 g. of mixed esters, b. p. 240—265°/3 mm., in 100 c.c. of ether). On treatment with bromine excess was just apparent when 3·65 c.c. had been added [Calc. for reaction (A), 5·00 c.c.; for reaction (B), 2·50 c.c.; for 45% of (A) and 55% of (B), 3·63 c.c.]. On the addition of water the dibromo-compound (VI) was precipitated, and from the filtrate, which behaved exactly as described for the *meso*-form, a few mg. of the dimethyl ester, m. p. 117°, were isolated.