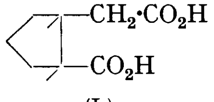
199. *Fused Carbon Rings. Part III. The Synthesis of* cis- *and* trans-cyclo*Pentane-*1*-carboxy-*2*-acetic Acids and the Stability of their Anhydrides.*

By A. H. Cook and R. P. Linstead.

It has been indicated in the preceding papers that the two forms (I and II) of *cyclo*pentane-1 carboxy-2-acetic acid are of importance both as reference compounds for the configurations of the *bicyclo*octanones and for comparison with their *cyclo*hexane analogues. Windaus, Hückel, and Reverey (*Ber.*, 1923, **56**, 91) have shown that the anhydrides of *cis*- and *trans*-hexahydrohomophthalic acids (III and IV) undergo interconversion at 240° to yield an equilibrium mixture containing 75% of the *trans*-anhydride, a fact which provided the first indication that the *trans*-locking of two six-membered rings could be more stable than the *cis*-.

It is now shown that both the corresponding *cyclo*pentane acids yield individual anhydrides which pass into an equilibrium mixture at 240°. *This contains* 86% *of the* cis-*isomeride,* which provides direct chemical evidence that a compound (such as VI), containing the *cyclo*pentane ring in a multiplanar form, is strained and comparatively unstable.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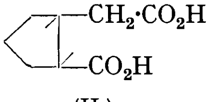
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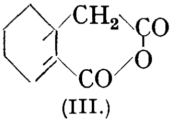
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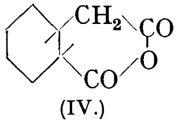
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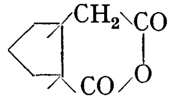
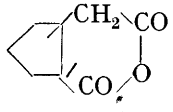
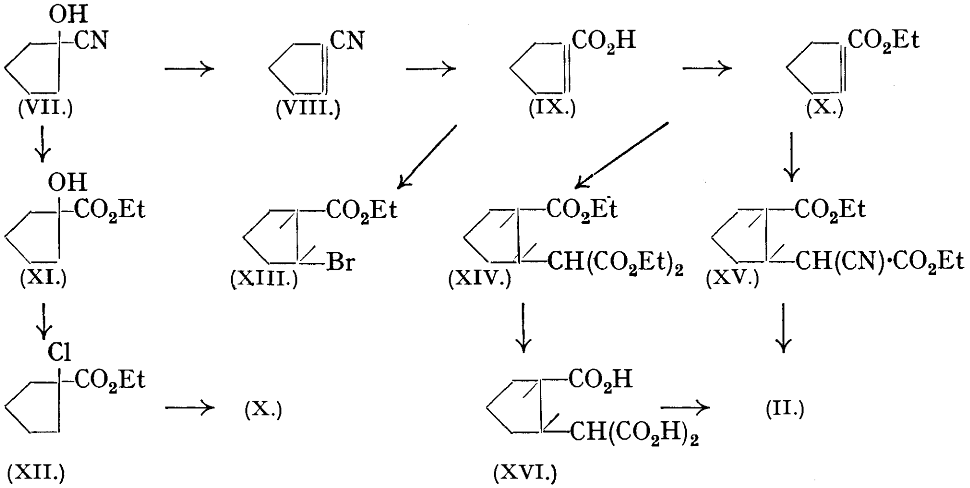




(VI.)

One synthesis of the acids (I) and (II) has already been described (p. 940), but it was necessary to investigate other preparative methods in view of the serious discrepancy between the melting points found by us (*cis*, 89° ; *trans*, 66°) and that (158—159°) recorded for the *trans*-acid by Sircar (J., 1927, 1255). We have accordingly made a general survey of the reactions indicated in the following scheme:



cyclo*Pentanone cyanohydrin* (VII), which has been used previously as a crude condensation product for synthetic purposes (Plant and co-workers, J., 1927, 484; 1928, 1906), has now been obtained in a pure condition both through the bisulphite compound and by Ultée's method. Unlike *cyclo*hexanone cyanohydrin (Ruzicka and Brugger, *Helv. Chim. Acta*, 1926, **9**, 399), it could not be dehydrated in the crude condition, but after distillation the action of thionyl chloride in benzene smoothly yielded Δ^1-*cyclo*pentenonitrile (VIII), which was hydrolysed by alkali to Δ^1-*cyclo*pentenecarboxylic acid (IX) and by alcoholic acid to the ester (X).

Sircar (*loc. cit.*) prepared the unsaturated ester (X) by hydrolysing the cyanohydrin to the hydroxy-acid, esterifying this, and dehydrating the ester (XI) by treatment with phosphorus pentachloride, followed by removal of hydrogen chloride with boiling diethylaniline. We find that the hydroxy-ester can be converted into the unsaturated ester by treatment with phosphorus pentachloride *alone* owing to the instability of the intermediate tertiary halide (XII). The yield is better if no base is used, and the ease of dehydration in this way contrasts with the difficulty experienced by v. Auwers and Krollpfeiffer (*Ber.*, 1915, **48**, 1394) in dehydrating the corresponding hydroxy-acid. The lability of the tertiary halogen, which is of general importance in the preparation of unsaturated acids or esters from ketones, does not appear to be generally recognised, although some analogies can be found (*e.g.*, Bredt's preparation of methyl dehydrocamphorate; *Annalen*, 1913, **395**, 26).

The unsaturated ester (X) prepared in all these ways (from the acid IX, by Sircar's method, and by treatment of XI with phosphorus pentachloride) yielded the same acid (IX) on hydrolysis. There is, however, reason to believe that Sircar's ester is not the pure Δ^α-isomeride, because it gives comparatively poor yields of addition products on treatment with ethyl sodiomalonate or sodiocyanoacetate, whereas the other esters behave normally. It is probable, therefore, that the use of diethylaniline leads to the formation of a good deal of Δ^β-ester, which is isomerised to the stable Δ^α-acid during hydrolysis.

The acid (IX) has many remarkable properties. Its melting point is very high (121°) for so simple an unsaturated acid—Wislicenus has suggested that it may be bimolecular, but this seems unlikely in view of its volatility—and it simulates aromatic properties in its resistance to additive reagents. Its low affinity for iodine is in harmony with the Δ^α-structure. No 2-bromo-*cyclo*pentane-1-carboxylic acid could be obtained from it by treatment with hydrogen bromide, although in alcoholic ether the bromo-ester (XIII) was obtained, which could also be prepared directly from the unsaturated ester. This bromo-ester was of no synthetic value owing to the lability of the halogen.

The addition of ethyl sodiomalonate to the unsaturated ester proceeded with exceptional ease, being almost complete in 30 minutes. The product, *ethyl* trans-cyclo*pentane-1-carboxylate-2-malonate* (XIV), did not yield a normal sodio-compound and attempts to condense it with chloroacetic ester (with the object of obtaining an intermediate capable of yielding a *bicyclo*octane derivative) were fruitless. Molecular sodium gave a red mass which on treatment with halogen esters yielded the malonic ester unchanged and it is possible that the metal reacts with the carbethoxyl rather than the malonic ester group (compare the papers of Scheibler referred to on p. 953). There may be some connexion between the non-formation of the normal sodio-compound and the rapidity of the Michael addition.

As one carbethoxyl group is directly attached to the ring and as the ester is formed in the presence of sodium ethoxide, it follows from the work of Hückel and Goth (*Ber.*, 1925, **58**, 447) that the carbethoxy-malonic ester (XIV) must have the *trans*-configuration. This provides an additional confirmation of the structures assigned to the *bicyclo*octanes on p. 949.

Controlled hydrolysis of (XIV) gave the corresponding malonic acid (XVI), whilst prolonged hydrolysis yielded *trans-cyclo*pentane-1-carboxy-2-acetic acid (II) of m. p. 66° identical with that of Linstead and Meade (p. 946). The same acid was obtained by the action of heat on the malonic acid (XVI).

Reference may now again be made to Sircar's preparation. This involved the condensation of the unsaturated ester (X) with ethyl sodiocyanoacetate to the cyano-dicarboxylic ester (XV), which was hydrolysed to a *trans*-acid reported to melt at 158°. On repeating this process the acid obtained was the *trans*-acid (II) of m. p. 66° identical with that prepared by the other methods. Sircar's work is therefore erroneous.*

The *trans*-acid readily yielded a crystalline *anhydride* (VI), which could be reconverted into the parent acid. At 240° the anhydride rapidly isomerised into a liquid anhydride, which on hydration gave a mixture from which pure *cis-cyclo*pentane-1-carboxy-2-acetic acid, m. p. 89°, could be separated. This was identical with the acid of Linstead and Meade.† The *cis*-acid regenerated the *trans*-acid on treatment with hydrochloric acid at 180°. The *cis*-acid readily gave a liquid *anhydride* (V) with acetyl chloride or acetic anhydride, which regenerated the *cis*-acid on hydration.

The two acids yielded *dianilides*, and the two anhydrides isomeric pairs of *anilic acids*, on treatment with aniline.

The interconversion of the anhydrides was followed quantitatively by thermal analysis. The equilibrated anhydride was converted into the corresponding mixture of acids, the melting point of which (82°) was compared with that of known mixtures. The same equilibrium mixture of anhydrides, which contained 86% of the *cis*-isomeride, was reached from both sides.

EXPERIMENTAL.

cyclo*Pentanone Cyanohydrin* (VII).—(1) The bisulphite compound from 100 g. of *cyclo*pentanone was cooled in ice and treated slowly, with vigorous shaking, with 86 g. of potassium cyanide in 150 c.c. of water. After 1 hour the upper layer of cyanohydrin was separated, the lower layer extracted with ether, the extract added to the upper layer, and the whole dried and distilled after the addition of concentrated sulphuric acid. Consistent yields of 15—20 g. of recovered ketone, 77 g. (65%) of cyanohydrin (b. p. 114°/14 mm.), and a small residue of the hydroxy-acid (corresponding to XI) were obtained. The freshly distilled *cyanohydrin* is pure (Found: C, 64·6; H, 8·4. C_6H_9ON requires C, 64·8; H, 8·5%), but in glass containers hydrogen cyanide is slowly evolved and the decomposition is rapid in the presence of bases, even ammonia. (2) Hydrogen cyanide from 250 g. of potassium cyanide and 500 c.c. of 50% sulphuric acid

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* Sircar's statement (*loc. cit.*) that the hydrolysis of the *cyclo*hexane ester corresponding to (XV) with hydrochloric acid gives at first a mixture of *cis*- and *trans*-hexahydrohomophthalic acids and only after 18 hours' boiling the *trans*-acid, can hardly be correct. The Michael addition must give a *trans*-product and boiling hydrochloric acid could not cause a configurational change. The mixture must have been due to incomplete decarboxylation.

† The common physical relationships between the acids (*i.e.*, *cis*-, lower melting point, more soluble) are reversed in these acids. Other exceptions to these usual rules are, however, known.

(vol.) was condensed in a freezing mixture, and 200 g. of ice-cold *cyclo*pentanone, mixed with 0·5 g. of caustic potash and a few drops of water, were added. After 6 hours at 0°, 10 drops of concentrated sulphuric acid were added, the excess of hydrogen cyanide was removed, and the residue distilled. Yield of cyanohydrin 230 g. (87%), b. p. 126°/26 mm.

Δ^1-cyclo*Pentenonitrile* (VIII).—A mechanically stirred mixture of 77 g. of the cyanohydrin and 105 c.c. of dry benzene was treated at 0° with 120 g. (1½ mols.) of thionyl chloride (dropwise, 75 minutes). The solution was then *gently* warmed for 1—1½ hours until hydrogen chloride was no longer vigorously evolved, cooled, and treated with ice, and the aqueous layer extracted with benzene. The combined benzene solutions were freed from hydroxy-acid and mineral acids by treatment with alkali, dried, and distilled. Δ^1-cyclo*Pentenonitrile* is a stable liquid with an odour resembling that of benzonitrile. Yield, 35—40 g.; b. p. 69°/15 mm., 81°/30 mm. (Found : C, 77·4; H, 7·5. C_6H_7N requires C, 77·4; H, 7·6%). The yield obtained by the Darzens process (thionyl chloride, pyridine, ether) was lower. The nitrile was not reduced by aluminium amalgam.

Δ^1-cyclo*Pentenecarboxylic Acid* (IX).—45 G. of the nitrile were hydrolysed for 36 hours with 60 g. of caustic potash in 600 c.c. of water. Acidification liberated the unsaturated acid (55 g., 90%) as an amorphous powder, becoming crystalline after an hour. The acid is very volatile in steam, but is best purified by crystallisation from hot water or petroleum or by sublimation. It melts at 121° and has little smell in the pure condition. The iodine addition (Linstead and May, J., 1927, 2565) was 1·0% in 10 minutes, 1·8% in an hour.

The acid in glacial acetic acid (1·5 g. in 8 c.c.) was saturated with hydrogen bromide at 0°. The volatile acids were removed over potash in a desiccator; the residue melted at about 65° (crude), but yielded only the unsaturated acid on recrystallisation. No addition of hydrogen bromide was observed to solutions of the acid in ether or suspensions in water. In ether containing a little alcohol ethyl 2-bromo*cyclo*pentanecarboxylate (see below) was formed.

Ethyl Δ^1-cyclo*Pentenecarboxylate* (X).—(1) The silver salt of the acid gave a comparatively poor yield of ester on treatment with ethyl iodide. (2) The acid was converted through the acid chloride (thionyl chloride) into the ester, b. p. 75°/10 mm.; excellent yield. (3) 50 G. of *cyclo*pentenonitrile were boiled vigorously for 20 hours with a mixture of 60 c.c. of sulphuric acid, 70 c.c. of absolute alcohol, and 70 c.c. of rectified spirit. The pasty mass was treated with water and extracted with ether; the extract was washed with sodium bicarbonate solution, and the residue from the ether distilled; yield, 55 g. (75%); b. p. 92°/25 mm. (Found : C, 68·4; H, 8·4. Calc. for $C_8H_{12}O_2$: C, 68·5; H, 8·5%). All the above samples yielded the acid of m. p. 121° on hydrolysis with hydrochloric acid. The preparation of the ester by the dehydration of ethyl *cyclo*pentanol-1-carboxylate is described below.

The unsaturated ester (50 g.) was treated with hydrogen bromide at 0° for 2 hours. The bromo-ester was freed from acid and distilled, 90% of (probably trans-) *ethyl* 2-*bromo*cyclo*pentane*-1-*carboxylate* being obtained, b. p. 126—127°/24 mm. (Found : Br, 36·2. $C_8H_{13}O_2Br$ requires Br, 36·3%). The lability of the bromine atom is shown in the following experiments. (1) The bromo-ester was boiled with an alcoholic suspension of potassium cyanide; ethyl *cyclo*pentenecarboxylate was recovered almost quantitatively. (2) A fine suspension of sodium cyanide made by the addition of anhydrous hydrogen cyanide to sodium ethoxide was treated with 42 g. of the bromo-ester and 0·5 g. of sodium iodide. After being boiled for 30 minutes, some 70% of unsaturated ester was obtained together with only 14% of a nitrogenous ester (b. p. 135—140°/24 mm.), probably ethyl 1-cyano*cyclo*pentane-2-carboxylate.

The unsaturated ester (X) was also prepared by Sircar's method as follows : *cyclo*pentanol-1-carboxylic acid, m. p. 103°, obtained in 80% yield by Meerwein and Unkel's method (*Annalen*, 1910, **376**, 156), was converted (compare *Ber.*, 1915, **48**, 1394) with boiling alcoholic sulphuric acid into the ester (XI) in 60—65% yield; b. p. 100—101°/23 mm. The hydroxy-ester (120 g.) was then slowly run on to 170 g. of phosphorus pentachloride. Phosphorus oxychloride was removed under reduced pressure, and the residue boiled for 3 hours with diethylaniline. The unsaturated ester was isolated from the product in the usual manner in 45% yield, b. p. 92°/24 mm. This process was greatly improved as follows : 70 g. of the hydroxy-ester were slowly dropped on 100 g. of phosphorus pentachloride, the phosphorus oxychloride removed under reduced pressure, and the light yellow residue left in contact with ice and water for 2 hours. The unsaturated ester was extracted, freed from mineral acids, and distilled; yield, 85%; b. p. 92°/25 mm.

Michael Additions.—(1) A mixture of 20 g. of the unsaturated ester (made by Sircar's method) and 20 g. of ethyl cyanoacetate was warmed (6 hours, steam-bath) with 3·5 g. of sodium in 40 c.c. of alcohol. The neutral fraction of the product yielded 10·5 g. (30%) of ethyl *cyclo*pentane-1-carboxylate-2-cyanoacetate (XV), b. p. 182—184°/12 mm. (2) Equimolecular quantities of the unsaturated ester (made by the PCl_5 method), malonic ester, and sodium ethoxide (200 c.c. of alcohol per mol.) were warmed on the steam-bath for 30 minutes; the product was treated with water, acidified, and extracted with ether, the extract washed with sodium bicarbonate solution and dried over sodium sulphate, and the residue after removal of ether was distilled. *Ethyl* trans-cyclo*pentane-1-carboxylate-2-malonate* (XIV) boiled at 181—182°/11 mm. Yield, 80—85% (Found : C, 59·8; H, 7·9. $C_{15}H_{24}O_6$ requires C, 60·0; H, 8·0%). A similar result was obtained by using the unsaturated ester made from the nitrile, but the yield obtained from the ester prepared by Sircar's method was poor. If heating was prolonged beyond 30 minutes, the yield was decreased.

The carbethoxy-malonic ester could not be condensed with ethyl chloroacetate by adding the reagent either to the crude Michael addition product or to the red sodio-compound formed from the pure ester and molecular sodium. Neither could ethyl *cyclo*pentenecarboxylate be induced to react to any extent with ethyl carboxysuccinate in the presence of sodium ethoxide.

Hydrolyses.—(1) The carbethoxy-malonic ester (XIV) was hydrolysed with 2 vols. of boiling concentrated hydrochloric acid until the oil had just disappeared (1—1½ hours). After removal of the mineral acid with the aid of a pump, the syrupy residue solidified to a mass of white needles of trans-cyclo*pentane-1-carboxy-2-malonic acid* (XVI) which, recrystallised from ethyl acetate–petroleum or, better, from concentrated hydrochloric acid, formed slender needles, m. p. 181·5°. The acid is insoluble in chloroform and petroleum, moderately soluble in ethyl acetate, benzene, alcohol and hydrochloric acid, and readily soluble in water [Found : C, 50·0; H, 5·6; equiv., 72·5. $C_9H_{12}O_6$ requires C, 50·0; H, 5·6%; equiv. (tribasic), 72·0]. When heated at its melting point for a few minutes, the acid evolved carbon dioxide vigorously; the residue soon solidified to the corresponding carboxy-acetic acid (II). (2) The carbethoxy-malonic ester was hydrolysed in the same manner, but for 12 hours. Removal of the mineral acid left the carboxy-acetic acid as an oil which rapidly solidified. (3) 10·5 G. of *cyclo*pentane-1-carbethoxy-2-cyanoacetic ester (made by Sircar's method) were boiled for 30 hours with 30 c.c. of concentrated hydrochloric acid. The mineral acid was removed under reduced pressure, and the organic acid extracted from the ammonium chloride by means of ether. Removal of the ether left the carboxy-acetic acid (II). trans-cyclo*Pentane-1-carboxy-2-acetic acid* (II) crystallised from a large bulk of light petroleum in clumps of small needles, m. p. 66° [Found : C, 55·5; H, 7·0; equiv., 86·0. $C_8H_{12}O_4$ requires C, 55·8; H, 7·0%; equiv. (dibasic), 86·0]. The same acid was obtained by all three methods given above and the product was identical with that of Linstead and Meade (p. 946).

6·5 G. of the acid were boiled for 30 minutes with 10 vols. of acetic anhydride and the excess of the reagent was removed under reduced pressure. The residue solidified over potash in a vacuum desiccator and was crystallised from light petroleum. The *anhydride* of trans-cyclo*pentane-1-carboxy-2-acetic acid* (VI) then separated in beautiful colourless detached needles, m. p. 43—44° (yield, almost theoretical). It is readily soluble in hot light petroleum, sparingly in cold (Found : C, 62·2; H, 6·5. $C_8H_{10}O_3$ requires C, 62·3; H, 6·5%). The same anhydride was obtained by the action of boiling acetyl chloride (10 vols., 1 hour). The anhydride was hydrolysed by cold dilute caustic soda or by water to the *trans*-acid, identified by m. p. and mixed m. p.

The *trans*-acid (1 g.) was converted by the action of phosphorus pentachloride (2·5 g.) in ether (15 c.c.) into the acid chloride, which with an excess of aniline yielded the *dianilide*. This separated from dilute alcohol in small needles, m. p. 214° (Found : C, 74·6; H, 6·9. $C_{20}H_{22}O_2N_2$ requires C, 74·7; H, 6·9%). The *trans*-anhydride (2 g.), dissolved in benzene (10 c.c.), was treated with 2·5 g. of aniline. After 36 hours in the cold the mixture was freed from benzene and dissolved in sodium carbonate solution. After removal of the unchanged aniline, acidification yielded a mixture (85%) of *trans*-anilic acids, $C_5H_8(CO_2H)\cdot CH_2\cdot CO\cdot NHPh$ and $C_5H_8(CO\cdot NHPh)\cdot CH_2\cdot CO_2H$, which was separated by fractional crystallisation from dilute alcohol. The less soluble *anilic acid* melted sharply at 130° (Found : C, 68·0; H, 6·8. $C_{14}H_{17}O_3N$ requires C, 68·0; H, 6·9%). The crude, more soluble acid was dissolved in the minimum quantity of hot benzene; a little of the less soluble form separated on cooling. The benzene was removed from the mother-liquor, and the residue crystallised from dilute alcohol, small plates of a second *anilic acid* being obtained, m. p. 108° (Found : C, 68·2; H, 6·8%).

15 G. of the *trans*-acid were refluxed for 24 hours with 40 c.c. of 5% alcoholic hydrogen chloride. The neutral product yielded 15·5 g. of *ethyl* trans-cyclo*pentane-1-carboxylate-2-acetate*, b. p. 148°/16 mm. (Found : C, 63·2; H, 8·8. $C_{12}H_{20}O_4$ requires C, 63·2; H, 8·8%). The

same ester was obtained in 90—95% yield through the silver salt. It did not condense with sodium and ethyl oxalate under Komppa's conditions.

Conversion into the cis-*acid.* After the *trans*-anhydride had been heated for 10 minutes at 240° (metal-bath), it failed to solidify on cooling, and hydration with dilute alkali, followed by acidification and extraction with ether, yielded an impure *cis*-acid, m. p. 81—83° (crude), 89° after crystallisation from hydrochloric acid. For preparative purposes it was better to distil the equilibrated anhydride (b. p. 174—175°/17 mm.; 6·3 g. from 10 g. of the *trans*-acid) and hydrolyse the yellow oil with just sufficient hot hydrochloric acid to effect solution. On cooling, the almost pure *cis*-acid separated (5·2 g.). *cis-cyclo*Pentane-1-carboxy-2-acetic acid crystallises from hydrochloric acid or from much petroleum in fine needles, m. p. 89° (it is only half as soluble in petroleum as the *trans*- isomeride) [Found: C, 55·6; H, 6·9; equiv., 86·2. Calc. for $C_8H_{12}O_4$: C, 55·8; H, 7·0%; equiv. (dibasic), 86·0]. The acid was identical with that of Linstead and Meade (p. 946). On treatment with hydrochloric acid at 180° it regenerated the *trans*-acid (m. p. 64—65°; 1·28 g. from 2·0 g.).

The pure *cis*-acid was boiled with 10 vols. of acetyl chloride for 30 minutes; removal of the reagent then left a liquid *anhydride* which failed to solidify in a vacuum desiccator after many weeks (Found: C, 62·0; H, 6·6. $C_8H_{10}O_3$ requires C, 62·3; H, 6·5%). It is undoubtedly a pure *cis*-anhydride, as it yields the pure *cis*-acid on hydrolysis and the derived anilic acids differ from those obtained from the *trans*-anhydride.

The cis-*dianilide*, prepared in the same manner as the *trans*-, crystallised from alcohol in short prisms, m. p. 234° (Found: C, 74·4; H, 6·9. $C_{20}H_{22}O_2N_2$ requires C, 74·7; H, 6·9%). Treatment of the *cis*-anhydride with aniline yielded a mixture of *anilic acids*, of which only that formed in preponderating amount was obtained pure. This crystallised from dilute alcohol in stellate clusters of needles, m. p. 115° (Found: C, 68·0; H, 7·05. $C_{14}H_{17}O_3N$ requires C, 68·0; H, 6·9%).

The two forms of *cyclo*pentane-1-carboxy-2-acetic acid are remarkably similar in general properties and no quantitative method for the separation of mixtures has been devised. The salts have very similar solubilities. The silver salts are amorphous, the calcium salts separate in crystalline form from boiling solutions, and the copper salts form sparingly soluble, light blue needles. The lead salts crystallise from dilute solutions in characteristic forms; that of the *cis*-acid in delicate single needles, that of the *trans*- in rosettes of short stout needles. The anhydrides absorb water at approximately the same rate and no separation could be achieved by "fractional hydration" in moist air. The composition of mixtures of the anhydrides was accordingly determined by hydration to the mixed acids and "thermal analysis."

Equilibration of Anhydrides.—A melting-point curve for mixtures of the pure acids was constructed as follows: The two acids were crystallised to constant melting point and dissolved in 20 parts of water. The requisite quantity of each solution was then measured out, mixed, and evaporated in a vacuum over phosphoric oxide. This method gave intimate mixtures of very small quantities of material. The m. p. of each mixture was taken (1) as it first separated; (2) after solidification and remelting; (3) after the mixture had been rubbed on porous earthenware and again dried over phosphoric oxide; and (4), as (3) but after solidification and remelting. All these m. p.'s were sharp, never differing by more than 1° and generally by less than 0·5°. The figures given in the table represent the mean of four readings to the nearest 0·5°.

Mixed melting points of cis- *and* trans-cyclo*pentane*-1-*carboxy*-2-*acetic acids.*

% *trans*-Acid	0	10	15	20	30	40	50	60	70	80	90	100
M. p.	89°	83·5°	81·5°	78·5°	73·5°	70·0°	65·0°	61·5°	55·5°	51·0°	53·5°	66°

The pure *trans*-anhydride was heated for 10 minutes at 240°, and the product hydrated with warm water for some time and then dried over phosphoric oxide. Found: m. p. 81·5° (mean), whence % *trans*-acid 14%. A mixture of the equilibrated material with a synthetic mixture containing 15% of the *trans*- acid melted at 81·5°. The *cis*- anhydride was heated for 5 minutes at 240° and examined similarly. M. p. 81·5° (mean); % *trans*-acid, 14%.

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