XVI.—*Studies in the Camphane Series. Part XVI.*
Camphorylcarbimide and Isomeric Camphorylcarb-
amides.

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THE readiness with which bornylamine, the amino-derivative of a fully hydrogenated cycloid, is converted into bornylcarbimide by the action of nitrous acid on the carbamide (Forster and Attwell, *Trans.*, 1904, **85**, 1188) has led us to examine the behaviour of more complex bases from the same standpoint. Choosing aminocamphor as a convenient type of the amino-ketones, we treated the hydrochloride with potassium cyanate in order to prepare the camphorylcarbamide obtained by Rupe (*Ber.*, 1894, **27**, 584, and 1895, **28**, 778), and at this stage encountered circumstances which have brought to light a camphoryl- ψ -carbamide exhibiting interesting properties.

Rupe describes the carbamide of aminocamphor as melting at 169° without evolving gas, and yielding with condensing agents camphorimidazolone, $C_{11}H_{16}ON_2$, infusible at 320° ; nitrous acid transformed his modification of camphorylcarbamide into a compound melting at 77° , the properties of which escaped examination, whilst the substance itself was not analysed.

Although we substantiate Rupe's account in every particular, our experiments show that a very slight modification of his conditions leads to an isomeric substance which melts somewhat indefinitely and decomposes at 184° , and yields with nitrous acid at 0° a true nitroso-derivative melting at 158° . The new compound, however, resembles Rupe's in furnishing camphorimidazolone when dissolved in concentrated sulphuric acid, and if treated with nitrous acid in the absence of precaution against rise of temperature, it gives the substance melting at 77° which was obtained by Rupe from his camphorylcarbamide; moreover, the differences in solubility, and in specific rotatory power, displayed by the two isomerides, are unimportant.

At first we suspected the discrepancy between Rupe's results and our own to depend upon a difference in the specimens of aminocamphor employed, because Rupe prepared his material by reducing *isonitrosocamphor* with zinc and acetic acid, whereas we applied the alkaline method, and in working with substitution products of camphor it is necessary to keep in view the possible occurrence of *cistrans*-isomerism, as in the case of bornylamine and neobornylamine. We find, however, that the formation of the isomeric camphorylcarbamides does not depend upon the source of the aminocamphor, but is influenced by the concentration of the liquid in which the hydrochloride of the base interacts with potassium cyanate, and by the presence of acid or alkali.

The production of two carbamide derivatives from a single base is a somewhat unusual occurrence, and the distinction offered by aminocamphor in this respect from bornylamine and aniline, each of which yields one carbamide only, appears to indicate the carbonyl group as causing the divergence of behaviour.

On considering the possible constitution of the two isomerides, numerous structural formulæ suggest themselves.

Of these expressions, II with IIa, IV, and VI are the tautomeric forms of I, III, and V, respectively, and although there can be little doubt that one or other of the compounds has the normal structure I, it is not easy to draw a definite conclusion regarding the isomeride; before doing so it will be necessary to indicate briefly the various properties which must be brought into agreement.

It has been mentioned that the substance melting at 77°, obtained by Rupe from camphorylcarbamide and nitrous acid, was not further examined by its discoverer; on preparing it for comparison with the nitroso-derivative of camphoryl- ψ -carbamide, we found that it is the

hitherto unknown camphorylcarbimide, $C_8H_{14} \begin{smallmatrix} \text{CH} \cdot \text{N} \cdot \text{C} \cdot \text{O} \\ | \\ \text{CO} \end{smallmatrix}$. Not only

does this compound arise from the *pseudo*-carbamide also if the action of nitrous acid is not controlled by a low temperature, but it is produced immediately, with liberation of nitrogen, when the freshly prepared camphorylnitroso- ψ -carbamide is warmed with water; if, however, the nitroso-compound is dried in the desiccator, or recrystallised from alcohol, boiling water has no action upon it. The behaviour of the two carbamides towards nitrous acid appears to differ, therefore, in the fact that a hydrated nitroso-derivative, forming an intermediate stage in the production of the carbimide, can be isolated only in the case of the *pseudocarbamide*.

Towards hydroxylamine acetate they behave alike. With this agent, both yield the oxime of camphorylcarbamide,

$C_8H_{14} \begin{smallmatrix} \text{CH} \cdot \text{NH} \cdot \text{CO} \cdot \text{NH}_2 \\ | \\ \text{C} \cdot \text{NOH} \end{smallmatrix}$, obtained from α -aminocamphoroxime hydro-

chloride and potassium cyanate by Lapworth and Harvey (Trans., 1902, 81, 553). Here again, the production of a single oxime from the two carbamides indicates the carbonyl group as the cause of isomerism, which disappears with oximation of the ketone.

If the hydrochloride of methylaminocamphor is treated with potassium cyanate, two carbamides again are formed. One of these has been described already by Duden and Pritzkow (Ber., 1899, 32, 1538) as melting at 185°; in addition to this compound we have obtained a *pseudo*-carbamide melting at 200°, and, as would be foreseen from the position of the methyl group, nitrous acid is without action on either compound. Of the two pairs of carbamides which have been studied, the one of lower melting point is transformed into the *pseudo*-modification by aqueous alkalis, whilst dilute acids bring about the converse change.

The foregoing facts lead us to regard Rupe's carbamide as having the normal structure I, and to represent the *pseudo*-modification by formula III. Expressions corresponding with V and VI have been used by Gabriel (Ber., 1889, 22, 2990; compare also Hirsch, Ber., 1890, 23,

$$\text{C}_8\text{H}_{14} \left\langle \begin{array}{c} \text{CH} \cdot \text{NH} \\ | \\ \text{C} = \text{N} \end{array} \right\rangle \text{CO},$$

By analogy with the camphorylcarbamides, the camphorylmethylcarbamide described by Duden and Pritzkow, and the *pseudo*-modification mentioned above, will be represented by the formulæ

EXPERIMENTAL.

precipitated aminocamphor was removed by means of ether, dissolved in 80 c.c. of 15 per cent. hydrochloric acid, and mixed with 50 grams of potassium cyanate in 150 c.c. of water. A viscous oil was immediately precipitated, and after 15 minutes the product was rendered alkaline with a 20 per cent. solution of sodium hydroxide, which transformed the oil into a crystalline paste in the course of 1 hour. The filtered substance was recrystallised from boiling water, concentrated solutions depositing leaflets, whilst needles separated from the diluted liquid; the specimen for analysis was further crystallised from benzene mixed with a small proportion of absolute alcohol, being thus obtained in felted masses of silky white needles which retained the solvent with such tenacity that until the substance had been heated for some time at 90°, the percentage of carbon was systematically higher than that indicated by theory.

0.1311 gave 0.3016 CO₂ and 0.1015 H₂O. C = 62.75; H = 8.66.

0.0990 „ 12.0 c.c. of nitrogen at 25° and 758 mm. N = 13.50.

C₁₁H₁₈O₂N₂ requires C = 62.86; H = 8.57; N = 13.33 per cent.

The melting point is somewhat indefinite, as the compound evolves gas, but fusion generally occurs at 184°, although one specimen crystallised from water melted at 188°. It is insoluble in boiling light petroleum, but is readily soluble in cold chloroform and absolute alcohol; warm ethyl acetate also dissolves it readily, but less freely than the isomeric carbamide, whilst warm acetone, in which it dissolves without difficulty, deposits it in well formed, rectangular plates, distinct from the silky needles in which camphorylcarbamide is deposited by the same medium. A solution containing 0.4609 gram in 25 c.c. of absolute alcohol gave $\alpha_D - 30'$ in a 2-dcm. tube, whence $[\alpha]_D - 13.5^\circ$.

Camphoryl- ψ -carbamide does not lose ammonia when boiled with 40 per cent. potassium hydroxide, but it is rapidly changed by concentrated sulphuric acid, yielding camphorimidazolone, which dissolves with difficulty in boiling alcohol, crystallising in lustrous needles infusible at 300°. The most satisfactory process for obtaining this compound is to dissolve 1 gram of the *pseudo*-carbamide in 2 c.c. of concentrated acid, heat in steam during 1 minute, and dilute largely with water; if the action is protracted, the colour of the liquid changes through pale red and intense reddish-brown to deep greenish-purple, finally acquiring the appearance of malachite green. These colour transformations are associated with the gradual destruction of the imidazolone.

Action of Hydroxylamine Acetate.—Two grams of the *pseudo*-carbamide were dissolved in 20 c.c. of absolute alcohol and heated with 4 grams of hydroxylamine hydrochloride and 10 grams of sodium acetate during 6 hours; on pouring the product into water, lustrous leaflets were

precipitated, crystallising from water in transparent, hexagonal plates. The oxime melts and decomposes at 202—203°, and appears to be identical with the carbamide obtained by Lapworth and Harvey from α -aminocamphoroxime (Trans., 1902, 81, 553).

In order to compare camphoryl- ψ -carbamide with Rupe's derivative, we prepared the latter by dissolving 10 grams of α -aminocamphor carbonate in 150 c.c. of 2 per cent. hydrochloric acid, and treating the filtered liquid with 10 grams of potassium cyanate; the filtrate remained clear during 24 hours, when long needles began to separate, and gradually filled the solution. The product, when completely dry, dissolved readily in cold benzene, but immediately separated in a voluminous mass of silky needles; this behaviour is doubtless due to assimilation of benzene of crystallisation, because the air-dried crystals require to be exposed to a temperature of 80° during a considerable period before the odour of the hydrocarbon is removed. In this respect the normal carbamide resembles the *pseudo*-modification, and with the exception already mentioned, the behaviour of the two substances towards solvents generally is exactly similar. Moreover, the colour-changes undergone by a solution of the carbamide in concentrated sulphuric acid on heating are identical with those recorded in connection with the *pseudo*-carbamide, and the oxime described above is obtained when the carbamide is heated with hydroxylamine acetate. Rupe's compound melts at 169°, and a solution containing 0.5275 gram in 25 c.c. of absolute alcohol gave a_D^{18} in a 2-dcm. tube, whence $[\alpha]_D + 7.1^\circ$.

In describing the production of this compound, Rupe (*Ber.*, 1895, 28, 778) mentions the formation of an oil when aminocamphor hydrochloride dissolved in water is warmed with potassium cyanate, and states that great difficulty was experienced in causing it to become crystalline. In the light of our experiments, it appeared probable that this oil was a mixture of camphoryl- ψ -carbamide with the normal compound, and having found that alkali facilitated its solidification, we proceeded to examine the behaviour of Rupe's carbamide towards alkali; 1 gram of the finely powdered substance was agitated with 10 per cent. sodium hydroxide during 2 hours, when the product was found to consist of the *pseudo*-carbamide. The converse change is brought about by dilute acids, a specimen of camphoryl- ψ -carbamide, when left in contact with dilute hydrochloric acid, and shaken at intervals during several days, changing completely into Rupe's compound.

Twenty grams of camphoryl-ψ-carbamide were suspended in 1 litre of water, treated with 60 c.c. of concentrated hydrochloric acid in 150 c.c. of water, and cooled by the addition of crushed ice; 20 grams of solid sodium nitrite were then added slowly, the liquid being stirred vigorously during the operation. After half an hour, the bulky, pale yellow precipitate was filtered, washed, dried in the desiccator, and subsequently crystallised from alcohol, separating in lustrous, pale yellow needles; when newly withdrawn from the mother liquor, it melts and decomposes at 142°, but after some days the crystals become opaque and melt at 158°.

0.1433 gave 0.2874 CO₂ and 0.0960 H₂O. C = 55.08; H = 7.44.

0.2170 „ 33.6 c.c. of nitrogen at 25° and 760 mm. N = 17.30.

C₁₁H₁₇O₃N₃ requires C = 55.23; H = 7.16; N = 17.57 per cent.

A solution containing 0.3138 gram dissolved in 25 c.c. of chloroform gave α_D 4° 15' in a 2-dem. tube, whence [α]_D + 169.3°.

The nitroso-derivative is insoluble in boiling light petroleum, and only sparingly soluble in benzene, from which it crystallises in silky needles; chloroform, ethyl acetate, and acetone dissolve it freely. It gives the Liebermann reaction with all the stages well defined, and does not reduce Fehling's solution on boiling.

In preparing the compound by the above process it is of the greatest importance to keep the liquid within a few degrees of zero, and to add the nitrite very slowly; if these precautions are not observed, nitrogen is evolved briskly, and the product consists of camphorylcarbimide (see below). It appears that the tendency to produce this substance is great, because even the nitroso-derivative, when freshly prepared, and while still wet, yields the carbimide on treatment with hot water; for this reason it is necessary to dry the substance thoroughly in the desiccator before attempting to re-crystallise it.

When the nitroso-derivative is treated with cold concentrated aqueous caustic potash, a very vigorous action occurs, nitrogen is liberated, and an oil produced which recalls the odour of geranium; we are still investigating this change, and find that the production of an acidic substance free from nitrogen is accompanied by elimination of cyanic acid. The reduction of the compound has also given interesting results, which we hope to communicate shortly.

Twenty grams of methylaminocamphor prepared by reducing the nitrogen methyl ether of *isonitrosocamphor* with zinc and acetic acid (Forster, *Trans.*, 1904, **85**, 898) were dissolved in 50 c.c. of 15 per cent. hydrochloric acid, and slowly treated with 30 grams of potassium cyanate dissolved in 50 c.c. of water; at first the liquid effervesced vigorously and remained clear, but when about half the salt had been added, a viscous oil separated. After an interval of 5 minutes, 10 per cent. sodium hydroxide was added, causing the oil to solidify rapidly, yielding a crystalline product which weighed 20 grams. The compound is sparingly soluble in boiling water, from which it crystallises in lustrous, prismatic needles, or brilliant leaflets, according to the concentration; it melts and evolves gas at 200°.

0·1639 gave 0·3846 CO_2 and 0·1360 H_2O . $\text{C} = 64·00$; $\text{H} = 9·22$.

$\text{C}_{12}\text{H}_{20}\text{O}_2\text{N}_2$ requires $\text{C} = 64·28$; $\text{H} = 8·99$ per cent.

A solution containing 0·8155 gram in 25 c.c. of absolute alcohol gave α_D^{27} in a 2-dcm. tube, whence $[\alpha]_D^{27} + 6·9^\circ$. The compound is very sparingly soluble in boiling light petroleum, but dissolves readily in hot benzene, crystallising in refractive, clear cut prisms; it is moderately soluble in cold ethyl acetate and acetone, crystallising from both in slender, prismatic needles. Absolute alcohol dissolves it readily, and the solution gives no precipitate with ammoniacal silver nitrate, but on boiling the liquid, reduction takes place slowly. The methyl-ψ-carbamide becomes yellow when boiled with 10 per cent. potassium hydroxide, but ammonia is not evolved; on boiling with Fehling's solution it remains unchanged. The substance distinguishes itself from camphoryl-ψ-carbamide by its indifference towards hydroxylamine acetate, and by its failure to yield a nitroso-derivative.

Action of Sulphuric Acid. — Camphorylmethyl-ψ-carbamide dissolves in hot dilute sulphuric acid, and in the cold concentrated acid with slight rise of temperature. If the latter solution is heated during several minutes in boiling water, the colour deepens from yellow to orange, pale red, intense brownish-red, and finally rich purplish-blue, resembling at this stage the colour developed by ferric chloride with enols. On dilution with water, the liquid yields no precipitate, but develops a green coloration, sufficiently intense at the later stages to recall malachite green. Wool is dyed in a green shade from this acid solution, but the colour produced is not permanent on washing, either with distilled or tap water; the fibre, however, retains a pale brown hue, the green colour being quickly

restored in its original intensity by soaking in dilute mineral acid, but not by acetic acid. If the deep green liquid is rendered alkaline, a pale brown solution is formed, in which acids develop the green colour; the chromophore is therefore amphoteric, and it is not extracted by ether from its solutions in acids and alkalis.

This description of camphorylmethyl- ψ -carbamide distinguishes the substance from the carbamide derivative prepared by Duden and Pritzkow (*Ber.*, 1899, **32**, 1542); these authors give no details relating to their method of preparation, but by the action of potassium cyanate on the hydrochloride of methylaminocamphor under the conditions favourable to the production of Rupe's camphorylcarbamide from aminocamphor, we have obtained the compound described by Duden and Pritzkow. Twenty grams of methylaminocamphor dissolved in 300 c.c. of 2 per cent. hydrochloric acid were treated with 25 grams of potassium cyanate in 50 c.c. of water; after half an hour, the filtered liquid began to deposit colourless crystals, the product, after 12 hours, weighing 17.5 grams. It crystallises from benzene in needles which retain the solvent with great tenacity, and, if previously heated for some time at 80° to remove benzene of crystallisation, it melts at 189°, evolving gas (Duden and Pritzkow give 185°). A solution containing 0.4823 gram in 25 c.c. of chloroform gave $\alpha_D - 58'$ in a 2-dcm. tube, whence $[\alpha]_D - 20.0^\circ$.

The two isomerides thus revealed behave alike towards ammoniacal silver nitrate and Fehling's solutions, and a very close examination is required to disclose differences in their solubility relations. Moreover, camphorylmethylcarbamide resembles the *pseudo*-modification in its indifference towards nitrous acid and by its failure to yield an oxime; the changes occurring in the colour of a solution in concentrated sulphuric acid correspond exactly with those displayed by the isomeride.

It has been mentioned that dilute sodium hydroxide transforms the camphorylcarbamide described by Rupe into the isomeric substance prepared by us, and we find that the two camphorylmethylcarbamides are related in the same fashion. One gram of Duden and Pritzkow's carbamide (m. p. 189°) was suspended in 10 per cent. sodium hydroxide, and shaken from time to time during 4 days; the product was then filtered, washed, and recrystallised from benzene, when it melted at 200°.

Twenty-five grams of Rupe's camphorylcarbamide were treated with nitrous acid under the conditions described for the preparation of camphorylnitroso- ψ -carbamide; brisk evolution of nitrogen took place, and addition of nitrite was continued until nitric oxide was liberated,

a white precipitate separating gradually. After an interval, this was filtered, washed, and dried in the desiccator. Some difficulty was experienced in purifying the compound, because it is very freely soluble in organic media, and very sensitive to the action of water. Bornylcarbimide can be distilled in steam without undergoing conversion into dibornylcarbimide to an extent greater than 10 per cent., but camphorylcarbimide, although readily volatile, changes quickly into dicamphorylcarbimide when treated with boiling water. Moreover, when the substance is warmed with alcohol, a very vigorous action takes place, but by dissolving it in cold absolute alcohol, diluting this with water, and filtering without delay the crystals which separate during the next few minutes, the carbimide may be obtained in silky, white needles melting at 77°.

0.1782 gave 0.4458 CO₂ and 0.1255 H₂O. C = 68.22; H = 7.87.

C₁₁H₁₅O₂N requires C = 68.39; H = 7.77 per cent.

A solution containing 0.4745 gram in 25 c.c. of chloroform gave $\alpha_D - 4.51'$ in a 2-dm. tube, whence $[\alpha]_D - 124.5^\circ$.

The carbimide may be obtained also by warming freshly prepared moist camphorylnitroso- ψ -carbamide with water, and even direct from the *pseudo*-carbamide by the action of nitrous acid if the operation is conducted without a liberal supply of ice. An attempt to prepare a nitroso-derivative of the oxime obtainable from the isomeric camphoryl-carbamides also gave rise to the carbimide. The oxime suspended in dilute hydrochloric acid was cooled with ice, and treated with its own weight of solid nitrite, which immediately developed a bright blue coloration; this is doubtless due to an unstable nitroso-derivative, because the filtrate from camphorylcarbimide quickly became turbid, lost its colour, and deposited a further quantity of the *isocyanate*, which is easily recognised by the intense, stupefying odour perceptible on warming the substance with water.

Camphorylcarbimide reacts most vigorously with organic bases, usually forming well-defined carbamides; in the case of aniline and phenylhydrazine, however, although action undoubtedly occurs, we have not succeeded in isolating a crystalline product.

Piperidine (1 mol.) diluted with benzene was added to a solution of camphorylcarbimide in the same solvent, when heat was developed, and crystals began to separate almost immediately. After 2 days the product was drained, recrystallised from dilute alcohol, and then from benzene, which deposited lustrous, transparent needles, exceeding

2 cm. in length; when freshly withdrawn from the mother liquor, these melted at 181° , but after exposure to air during 24 hours they became opaque, and melted at 186° .

0.1438 gave 0.3642 CO_2 and 0.1230 H_2O . $\text{C} = 69.08$; $\text{H} = 9.50$.

$\text{C}_{16}\text{H}_{26}\text{O}_2\text{N}_2$ requires $\text{C} = 69.05$; $\text{H} = 9.35$ per cent.

A solution containing 0.4000 gram in 25 c.c. of chloroform gave $\alpha_D 1^{\circ}17'$ in a 2-dcm. tube, whence $[\alpha]_D 40.1^{\circ}$. The compound is insoluble in boiling water, and sparingly soluble in boiling light petroleum, but it dissolves readily in alcohol, acetone, ethyl acetate, chloroform, and hot benzene.

Bornylamine (1 mol.) dissolved in benzene was added to camphorylcarbimide in the same solvent. Considerable development of heat occurred, but no crystals separated until light petroleum was added, precipitating minute, colourless granules. On redissolving in benzene, and adding petroleum until the liquid became turbid, lustrous, white needles separated slowly. The derivative melts and decomposes at 305° .

0.1457 gave 0.3880 CO_2 and 0.1318 H_2O . $\text{C} = 72.62$; $\text{H} = 10.05$.

$\text{C}_{21}\text{H}_{34}\text{O}_2\text{N}_2$ requires $\text{C} = 72.83$; $\text{H} = 9.83$ per cent.

A solution containing 0.2987 gram dissolved in 25 c.c. of chloroform gave $\alpha_D 41'$, whence $[\alpha]_D = 28.6^{\circ}$. The carbamide is slightly soluble in boiling light petroleum, but the crystals which separate are indefinite; it dissolves readily in benzene, ethyl acetate, alcohol, and hot acetone, crystallising from the last named in aggregates of prismatic needles.

Camphorylcarbimide was suspended in water and heated in a reflux apparatus until the penetrating odour characteristic of the substance was no longer perceptible; the product was filtered, and crystallised from dilute alcohol, which deposited aggregates of lustrous needles melting at 261° .

0.1157 gave 0.2952 CO_2 and 0.0924 H_2O . $\text{C} = 69.54$; $\text{H} = 8.87$.

$\text{C}_{21}\text{H}_{32}\text{O}_3\text{N}_2$ requires $\text{C} = 70.00$; $\text{H} = 8.88$ per cent.

A solution containing 0.5020 gram dissolved in 25 c.c. of chloroform gave $\alpha_D 1^{\circ}32'$ in a 2-dcm. tube, whence $[\alpha]_D = 38.2^{\circ}$. It is insoluble in boiling water, and sparingly soluble in boiling light petroleum,

from which it crystallises in silky needles on cooling; acetone, ethyl acetate, alcohol, and chloroform dissolve it freely, whilst benzene dissolves it readily when hot, a felted mass of needles being deposited on cooling.

Methyl Camphorylcarbamate (Camphorylmethylurethane),

Five grams of camphorylcarbimide were dissolved in 20 c.c. of pure methyl alcohol, and allowed to remain several weeks in the desiccator; as the alcohol evaporated, a transparent, gummy syrup was deposited, in which a few small needles were embedded. On dissolving this product in alcohol, and adding water until the solution became turbid, dicamphorylcarbamide was precipitated, but the filtrate yielded slender white needles of the methylurethane, which melts at 108° after recrystallisation from petroleum.

0.2040 gave 0.4779 CO_2 and 0.1566 H_2O . $\text{C} = 63.89$; $\text{H} = 8.53$.

$\text{C}_{12}\text{H}_{19}\text{O}_3\text{N}$ requires $\text{C} = 64.00$; $\text{H} = 8.44$ per cent.

A solution containing 0.2636 gram dissolved in 25 c.c. of chloroform gave $\alpha_D 55'$ in a 2-dcm. tube, whence $[\alpha]_D 43.5^\circ$.

The ethylurethane, prepared as in the case of the foregoing substance, separated from the dilute alcoholic filtrate in large, transparent prisms melting at 88° .

0.1714 gave 0.4102 CO_2 and 0.1413 H_2O . $\text{C} = 65.27$; $\text{H} = 9.18$.

$\text{C}_{13}\text{H}_{21}\text{O}_3\text{N}$ requires $\text{C} = 65.27$; $\text{H} = 8.78$ per cent.

A solution containing 0.5262 gram in 25 c.c. of chloroform gave $\alpha_D 1^\circ 38'$ in a 2-dcm. tube, whence $[\alpha]_D = 38.3^\circ$. Both urethanes are readily soluble in organic media, excepting light petroleum, and display a marked tendency to remain superfused.

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