261. *The Reaction between Acetone and Ammonia : The Formation of Pyrimidine Compounds Analogous to the Aldoxans of Späth.*

By R. B. Bradbury, N. C. Hancox, and H. H. Hatt.

The reaction of acetone with ammonia can be conducted to give 90% yields of the monohydrate of 2 : 2 : 4 : 4 : 6-pentamethyl-2 : 3 : 4 : 5-tetrahydropyrimidine, which is readily dehydrated to the anhydrous base. By reduction with sodium in alcohol it gives the corresponding hexahydropyrimidine, and with aluminium amalgam an open chain primary-secondary diamine, $C_9H_{22}N_2$. The hexahydropyrimidine is hydrolysed with extreme ease to 2 : 4-diamino-2-methylpentane and acetone.

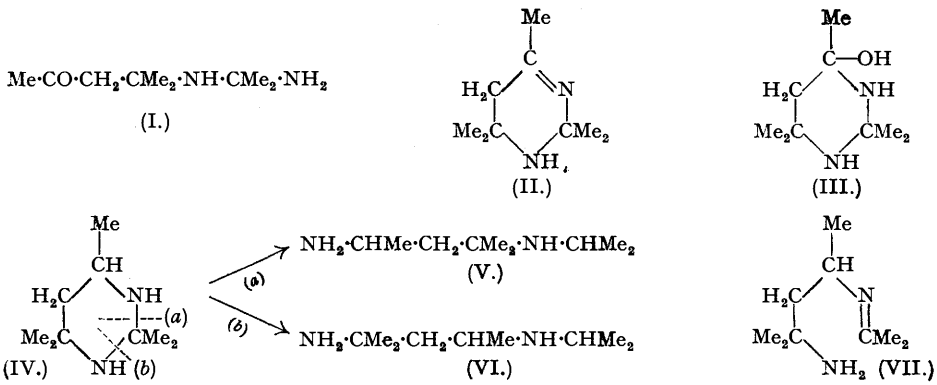
The mechanism of the formation of (II) is discussed in the light of its synthesis from mesityl oxide, acetone, and ammonia, and its quantitative hydrolysis to diacetonamine.

Mesityl oxide and ammonia can be condensed to produce a substituted tetrahydropyrimidine analogous in structure to paraldol.

DIACETONAMINE and triacetonamine can both be obtained from the reaction of acetone with ammonia followed by acidification of the reaction products, and Heintz (*Annalen*, 1874, **174**, 133) first showed that triacetonamine was produced if the reaction mixture was heated, but that at room temperature diacetonamine was the chief product. The preparation of these compounds has since been improved by the use of calcium chloride to hasten the absorption of ammonia and to assist in the removal of water (Everest, *J.*, 1919, **115**, 588; Francis, *J.*, 1927, 2897). Further Suzuki and Horie (*Bull. Inst. Phys. Chem. Res. Japan*, 1932, **11**, 383) have found that the formation of diacetonamine was catalysed by ammonium salts, especially the nitrate and nitrite. Other compounds have been obtained from this reaction. Hock and Stuhlmann (*Ber.*, 1928, **61**, 470) by working at low temperatures isolated a compound C_3H_9ON which, as it decomposed at room temperatures into acetone and ammonia, they regarded as the initial product of the reaction analogous to aldehyde-ammonia. Patterson and McMillan (*J.*, 1921, **119**, 267) found that after a protracted reaction period a compound $C_9H_{20}ON_2$ could be obtained by cooling, to which they assigned structure (I). It has not hitherto been further investigated.

We have now found that, by the use of ammonium chloride and calcium chloride as catalysts, this compound is formed in 90% yield from acetone in 24 hours. From examination of its properties we conclude that it is a *hydrate* of 2 : 2 : 4 : 4 : 6-*pentamethyl*-2 : 3 : 4 : 5-*tetrahydropyrimidine* (II) to which it can be readily dehydrated over sodium hydroxide or sulphuric acid, or by distillation alone or with benzene as entrainer. The anhydrous base readily absorbs water to re-form the hydrate, and the facility of these changes is evidence for the hydrate structure assigned rather than the possible alternative (III). It seems possible that (II) may be identical with the compound $C_9H_{18}N_2$ named "acetonin" by Städeler (*Annalen*, 1859, **111**, 305; Mulder, *ibid.*, 1873, **168**, 229).

The base (II) is readily hydrolysed by boiling water to mesityl oxide, acetone, and ammonia, and by oxalic acid to ammonium and diacetonamine hydrogen oxalates and acetone. The observed molecular refraction (47·8) of the anhydrous base agrees well with the value calculated for structure (II) (47·84). Further support to this structure is given by the observation that both (II) and its hydrate are reduced by sodium and alcohol to 2 : 2 : 4 : 4 : 6-*pentamethylhexahydropyrimidine* (IV) and that a *diamine* of structure (V) or (VI) is produced by the action of aluminium amalgam on (II), its hydrate, or (IV).



The pentamethylhexahydropyrimidine (IV) is hydrolysed more readily than the parent hexahydropyrimidine, first described by Titherley and Branch (*J.*, 1913, **103**, 330), yielding 2 : 4-diamino-2-methylpentane and acetone rapidly with dilute acids and slowly with water alone. Although it is readily converted into open chain compounds, there is no evidence for the co-existence of the Schiff's base (VII), for, unlike hexahydropyrimidine, (IV) distils without the formation of a resinous polymer and the observed molecular refraction (48·7) agrees closely with that calculated for (IV) (48·77).

Ease of ring fission in the methyl-substituted hydropyrimidines doubtless accounts in part for failure to prepare acyl derivatives, which the presence of imino- and hydroxyl groups in (II), (III), and (IV) suggests as possible. Titherley and Branch (*loc. cit.*) obtained the dibenzoyl derivative of hexahydropyrimidine by normal Schotten–Baumann procedure, but from (II) and its hydrate we obtained only *N*-benzoyldiacetonamine and from (IV) only 2 : 4-*dibenzamido*-2-

methylpentane. 4 : 4 : 6-*Trimethylhexahydropyrimidine* has been synthesised and shows intermediate properties, giving a little of its *dibenzoyl* derivative together with much 2 : 4-dibenzamido-2-methylpentane. Failure to acylate can be viewed as evidence for the hydrate structure rather than (III), for the imino-group in (II) is sterically almost identically placed with the imino-group of triacetonamine, which forms a hydrate and a nitroso-derivative, but so far has not been benzoylated. Actually (II) reacts with nitrous acid and gives a compound of the expected composition, $C_9H_{17}ON_3$, but the yield is small and the properties are abnormal.

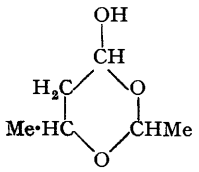
The hydrate of (II) can by synthesised from mesityl oxide, acetone, and ammonia, thus reversing the hydrolysis with acids and suggesting that formation from acetone proceeds by way of diacetonamine and not through the hypothetical intermediate, $CMe_2(NH_2)\cdot CMe_2\cdot OH$, proposed by Patterson and McMillan. The preparation of diacetonamine from acetone and ammonia by the method of Everest therefore involves the formation of the hydrate, followed by its hydrolysis with oxalic acid, the scheme being :

$$\text{Acetone} + \text{Ammonia} \longrightarrow \text{Diacetonamine} \xrightarrow[\text{ammonia}]{\text{Acetone and}} \text{Hydrate of (II)} \xrightarrow[\text{acid}]{\text{Oxalic}} \text{Diacetonamine.}$$

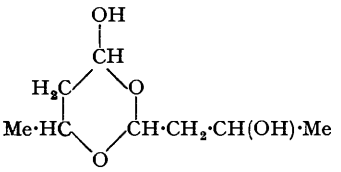
Presumably (II) is also an intermediate in the formation of triacetonamine.

The strong catalytic action of ammonium salts in the formation of (II) can be attributed to their acidic character in a mixture of acetone and ammonia. In support of this view, trimethylammonium chloride, which can still donate a proton, exhibits a like powerful catalytic action, whereas tetramethylammonium chloride, being unable to react in this way, has an action even weaker than calcium chloride. These catalytic effects must apply mainly to the initial formation of diacetonamine, for the hydrate of (II) is formed readily from diacetonamine, acetone, and ammonia in absence of a catalyst.

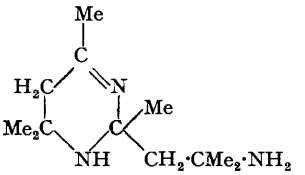
An analogy is apparent between the suggested mechanism for the reaction between acetone and ammonia and that given by Späth for the formation of aldoxan (VIII) by condensation of acetaldehyde (*Ber.*, 1943, **76**, 57). The reaction between mesityl oxide and ammonia provides a further analogy. When the ammonia used is in excess of that needed to form diacetonamine, a compound of the formula $C_{12}H_{25}N_3$ forms slowly. To this is assigned structure (X), closely analogous to the structure (IX) determined by Späth for paraldol. Like the substituted



(VIII.)



(IX.)



(X.)

tetrahydropyrimidine (II), this 2 : 4 : 4 : 6-*tetramethyl*-2-(2-*amino*isobutyl)-2 : 3 : 4 : 5-*tetrahydropyrimidine* is readily reduced to the corresponding substituted *hexahydropyrimidine*. It forms no hydrate, but, like (II), is unstable in the presence of acids, which cause reversal of the above synthesis and give ammonium and diacetonamine hydrogen oxalates as the chief products of decomposition.

Diacetonamine is conveniently prepared from the hydrate of (II), a yield of 93% being obtained by boiling with alcoholic oxalic acid. This represents a yield of 56% based on the acetone used to make the hydrate and compares favourably with the 37—42% reported by Everest (*loc. cit.*), in which allowance was made for recovered acetone, and with the 70—75% yields from mesityl oxide (*Org. Synth.*, VI, 28; Smith and Adkins, *J. Amer. Chem. Soc.*, 1938, **60**, 407). The synthesis of 2 : 4-diamino-2-methylpentane from acetone through the hydrate of (II) and (IV) comprises three steps all with yields exceeding 80%, and is preferable to the preparation from acetone proceeding through mesityl oxide, diacetonamine, and its oxime.

Experimental.

(All melting points are corrected.)

2 : 2 : 4 : 4 : 6-*Pentamethyl*-2 : 3 : 4 : 5-*tetrahydropyrimidine Hydrate*.—(*a*) Acetone (290 g., 5 mol.), calcium chloride (" A.R. dried ", 40 g.), and ammonium chloride (10 g.) were cooled in an autoclave to — 40°, and liquid ammonia (110 g., 6·5 mol.) was added. After 24 hours' stirring at room temperature, the solid product was melted by gentle warming (40°), and the top layer separated, allowed to solidify,

and dried on porous plates (yield, 252 g.; 88%). This material is almost pure, but the small amounts of ammonium and calcium chlorides present can be removed by crystallisation from ether.

The amounts of ammonia, ammonium chloride, and calcium chloride stated above are the least for a good yield, and the use of larger amounts does not perceptibly affect the yield. When the reaction time is prolonged to 3 days the yield is increased by 1—2%.

With acetone and ammonia alone the reaction mixture remains liquid for months, although some hydrate is formed and crystallises out on cooling to − 40°. Thus after 7 days a 17% yield is obtained. In presence of ammonium chloride the hydrate forms rapidly, but the solid reaction product does not separate into two layers when melted, and removal of ammonium chloride requires extraction with ether. With calcium chloride alone the yield is smaller even with a longer reaction period. The product, however, forms two liquids; the lower, containing the calcium chloride and water, is easily removed, and the upper when cooled to − 40°, deposits the hydrate. The presence of small amounts of lime in the calcium chloride does not affect the yield. The presence of water is beneficial; thus fused calcium chloride alone and with half its weight of water gave yields of 55 and 65% respectively.

The table summarises the results of experiments in which 290 g. (5 mols.) of acetone and 110 g. (6·5 mols.) of ammonia were used. The reaction took place at room temperatures and the amount of catalyst used was always equivalent to 10 g. of ammonium chloride.

Catalyst.	Calcium chloride (g.).	Time (days).	Yield of (II) (%).	Catalyst.	Calcium chloride (g.).	Time (days).	Yield of (II) (%).
None	None	7	17	$NHMe_3Cl$	40	1	85
,,	40	4	65–70	NMe_4Cl	40	1	35
NH_4Cl	none	4	70	NMe_4Br	40	4	66
,,	40	1	88	$NHMe_3Cl$	none	4	63
NH_4NO_3	40	1	85	NMe_4Cl *	,,	4	35
NH_4Br	40	1	85				

* Added as a 55% aqueous solution.

(*b*) Mesityl oxide (120 g., 1·2 mol.) and ammonia (26 g., 1·5 mol.) were placed in an autoclave cooled to − 40° and stirred for 3 days at room temperature. The mixture was again cooled, acetone (71 g., 1·2 mol.) and ammonia (40 g., 2·4 mol.) were added and the stirring was continued for 4 days. The solid hydrate of (II) was purified by crystallisation from ether (yield, 139 g.; 66%).

The *hydrate* when freshly crystallised from ether forms colourless crystals, m. p. 43—44° [Found: C, 62·4; H, 11·5; N, 16·3; H_2O (by Dean–Stark), 10·5; equiv. (titration), 87. $C_9H_{18}N_2,H_2O$ requires C, 62·7; H, 11·7; N, 16·3; H_2O, 10·5%; equiv., 86]. In air it slowly becomes yellow and decomposes, liberating ammonia, but it can be kept for a considerable time in a stoppered vessel in a refrigerator. It is extremely volatile and sublimes to give large, well-formed crystals of hexagonal habit. It is soluble in cold water (70 g. per 100 ml.), in cold ether (19 g. per 100 ml.), and in most organic solvents.

The *oxalate* was prepared from equimolecular quantities of the hydrate (II) and anhydrous oxalic acid in ether (Found: C, 44·5; H, 7·1; N, 8·0. $C_9H_{20}ON_2,2H_2C_2O_4$ requires C, 44·3; H, 6·8; N, 8·0%).

When to the hydrate (II) and an equivalent amount of aqueous sodium nitrite at 0°, two equivalents of 2N-hydrochloric acid were added, reaction occurred with deposition of a colourless crystalline *substance* during 3 hours (yield, 10—15%), m. p., 168—170° (decomp.) (from acetone). The low yield, high m. p., and decomposition with dilute mineral acid without liberation of nitrous acid conflict with the supposition that it is the nitroso-derivative of (II), although the analytical data are in agreement. Molecular weight determinations gave double the expected values [Found: C, 59·2; H, 8·8; N, 22·8; *M* (in nitrobenzene), 371. $C_9H_{17}ON_3$ requires C, 59·0; H, 9·3; N, 23·0%; *M*, 183).

Diacetonamine Hydrogen Oxalate.—The crude hydrate of (II) (269 g.) prepared from acetone (290 g.) was dissolved in alcohol (300 ml.). An aliquot (5 ml.) was titrated with oxalic acid to determine the amount required (twice that needed for neutralisation: 365 g.). In the titration it is necessary to add about 3/4ths of the estimated amount of acid and then to warm to 80° for a few minutes to complete the hydrolysis of the hydrate. Titration at room temperature as directed by Everest gives low results and leads to the use of insufficient oxalic acid. The oxalic acid was dissolved in alcohol (2000 ml.), stirred vigorously, and the solution of the hydrate added in ½ hour at 30° or less. After a further ½ hour, 100 ml. of the solvent were removed and the ammonium hydrogen oxalate was filtered from the hot solution and extracted thrice with 300 ml. portions of boiling alcohol. The combined filtrates were cooled rapidly, and after 3 hours the diacetonamine hydrogen oxalate was collected and dried (m. p. 123—125°). The mother liquors, on evaporation, yielded a further 20 g. Yield, 309 g.; 56% based on the acetone taken. The maximum yield on this basis is 66·7%.

2 : 2 : 4 : 4 : 6-*Pentamethyl*-2 : 3 : 4 : 5-*tetrahydropyrimidine* (II).—Distillation of the hydrate under reduced pressure or dehydration using benzene as an entrainer, gives a liquid from which the *base* (II) is obtained by fractionation under reduced pressure (yield, 80%), b. p., 171° (decomp.)/774 mm., 55°/10 mm., 85°/50 mm.; $d_4^{25°}$ 0·8769; $n_D^{25°}$ 1·4561; $[R_L]_D$ 47·8 (Calc., 47·84) (Found: C, 70·3; H, 11·3; N, 18·5; equiv., 77. $C_9H_{18}N_2$ requires C, 70·1; H, 11·7; N, 18·2%; equiv., 77). The liquid is pale yellow and of ammoniacal odour. Addition of water to (II) gave the hydrate in 72% yield. There is some decomposition during the formation of (II), the water removed is strongly ammoniacal, and materials boiling higher than (II) are formed.

Both bases are readily decomposed in aqueous solution. The hydrate (1·72 g.) when boiled with an aqueous solution of oxalic acid (2·52 g.), the water removed on a steam-bath under slightly reduced pressure, and the dry residue extracted with absolute alcohol left ammonium hydrogen oxalate (1·03 g.; theory, 1·07 g.). The alcoholic solution on evaporation gave crude diacetonamine hydrogen oxalate, m. p., 124°, after crystallisation. In another experiment 1·72 g. were boiled with excess of aqueous oxalic acid and the acetone distilling was absorbed in water and estimated by the method of Messenger (Found: 0·57 g. Calc.: 0·58 g.). Attempts were made to benzoylate the hydrate by normal

Schotten–Baumann procedure, in dry pyridine, and in ether in presence of potassium carbonate. The only products identified were benzamide and *N*-benzoyldiacetonamine [m. p. (from alcohol) and mixed m. p. 99—100°].

2 : 2 : 4 : 4 : 6-*Pentamethylhexahydropyrimidine* (IV).—The hydrate (86 g.) was reduced with sodium (40 g.) in alcohol (250 ml.) and the product was first distilled with the alcohol and then separated by fractional distillation. The pure *product* (64 g., 82%) was colourless fuming liquid of sweet, slightly ammoniacal odour. It absorbs carbon dioxide from air and is hygroscopic; b. p., 56·5°/10 mm., 90·5°/50 mm., 172—175° (decomp.)/765 mm.; $d_{4^\circ}^{25^\circ}$ 0·8653, $n_D^{25^\circ}$ 1·4517; $[R_L]_D$ 48·7 [Calc. for (IV), 48·77; for (VII), 49·86] (Found: C, 69·3; H, 12·8; N, 18·2; equiv., 77. $C_9H_{20}N_2$ requires C, 69·3; H, 12·8; N, 18·0%; equiv., 78). Reduction of (II) also yields (IV), but preparation from the hydrate is preferable. Hydrolysis of (IV) with hot water is rapid and in an hour 80% of the calculated amount of acetone was liberated.

Conversion of (IV) *into* 2 : 4-*Diamino-2-methylpentane* (XI).—(IV) (54 g.) was added slowly to 2N-hydrochloric acid (380 ml.) at 0°, and after a day at room temperature the mixture was heated to remove acetone. Sufficient solid sodium hydroxide was added to give a 40% aqueous solution, the diamine layer was separated, and the aqueous liquors were extracted with ether. The *diamine* was dried over potassium hydroxide and barium oxide. Fractional distillation gave a colourless liquid (yield, 33·5 g.; 83%), b. p. 45—46°/10 mm.; $d_{4^\circ}^{25^\circ}$ 0·8312; $n_D^{25^\circ}$ 1·4386; $[R_L]_D$ 36·8 (Calc., 36·75) (Found: C, 62·6; H, 13·5; N, 23·1. $C_6H_{16}N_2$ requires C, 62·1; H, 13·8; N, 24·1%). (XI) was also prepared from diacetonamine by the method of Harries and Adamiantz (*Ber.*, 1901, **34**, 301); b. p. 42—46/10 mm.; $n_D^{18^\circ}$ 1·439; $d_{4^\circ}^{25^\circ}$ 0·836.

The *diacetyl* derivative obtained from (XI) and either acetic anhydride or keten formed colourless crystals from chloroform, m. p., 163° (Found: C, 60·0; H, 10·0; N, 14·2. $C_{10}H_{20}O_2N_2$ requires C, 60·0; H, 10·0; N, 14·0%). The *dibenzoyl* derivative obtained by the Schotten–Baumann procedure from (XI) formed colourless rosettes from alcohol, m. p., 153° (Found: C, 74·2; H, 7·4; N, 8·7. $C_{20}H_{24}O_2N_2$ requires C, 74·1; H, 7·4; N, 8·6%); it showed no depression of melting point with the derivative prepared from the specimen of (XI) synthesised from diacetonamine. This same dibenzoyl derivative is slowly formed when (IV) is benzoylated.

2 : 4-Diamino-2-methylpentane (XI), chloro-2 : 4-dinitrobenzene, sodium acetate, and alcohol were refluxed for 1 hour. The *bis*-2 : 4-*dinitrophenyl* derivative formed yellow crystals from *o*-dichlorobenzene, m. p., 235° (Found: C, 48·3; H, 4·4; N, 18·9. $C_{18}H_{20}O_8N_6$ requires C, 48·2; H, 4·5; N, 18·7%). With like experimental conditions (IV) gave this same compound.

2-*Keto*-4 : 4 : 6-*trimethylhexahydropyrimidine*.—Equimolecular amounts of 2 : 4-diamino-2-methylpentane and urea were heated at 150° for 1 hour. The *product* separated from acetone as white crystals, m. p. 205—206° (Found: C, 59·5; H, 9·7; N, 19·9. $C_7H_{14}ON_2$ requires C, 59·2; H, 9·9; N, 19·7%).

4 : 4 : 6-*Trimethylhexahydropyrimidine* (XII).—The method used by Titherley and Branch (*J.*, 1913, **103**, 334) for hexahydropyrimidine was adopted, and 2 : 4-diamino-2-methylpentane (XI) (2·09 g.) was neutralised with 0·2N-hydrochloric acid, an equal weight of (XI) added, followed by 36% aqueous formaldehyde (3·5 ml.) in 3 hours. After a further 2 hours, potassium hydroxide (60 g.) was added, the *product* extracted with ether, and the solution dried (BaO) and distilled. Yield, 3·3 g.; b. p. 55—57°/10 mm.; $n_D^{18^\circ}$ 1·457 [Found: C, 65·7; H, 12·8; N, 21·7; equiv., 132. $C_7H_{16}N_2$ requires C, 65·7; H, 12·5; N, 21·9%; equiv. (monoacid), 128].

Trimethylhexahydropyrimidine (XII) by Schotten–Baumann procedure, gave its own *NN'*-dibenzoyl derivative, together with 2 : 4-dibenzamido-2-methylpentane. The former being the more soluble, only a partial separation was made by fractional crystallisation from alcohol, and the final separation was made by hand picking. This *dibenzoyl* derivative forms needles, m. p. 156° (Found: C, 75·0; H, 6·9; N, 8·3. $C_{21}H_{24}O_2N_2$ requires C, 74·8; H, 7·1; N, 8·3%).

2 (*or* 4)-*Amino*-4 (*or* 2)-iso*propylamino*-2-*methylpentane* (V) *or* (VI).—This was obtained by reduction of (II), its hydrate, or (IV) with an equal weight of aluminium amalgam in moist ether. The reaction proceeded slowly and was complete after 2 days; the ethereal liquors were then filtered off, and the oxide sludge was washed with ether. When distilling the ethereal liquors the alcohol introduced with the amalgam distilled as a fore-fraction containing much diamine. Yields of pure *diamine* were 60—70% when this fore-fraction was neglected; with recovery from it the yields rose to 85%. Dried over sodium, (VI) forms a colourless hygroscopic liquid, b. p. 178°/760 mm. (no decomp.), 65—67°/10 mm., 104°/65 mm.; $d_{25^\circ}^{25^\circ}$ 0·8130; $n_D^{25^\circ}$ 1·432 (Found: C, 68·4; H, 14·0; N, 17·7; equiv., 81. $C_9H_{22}N_2$ requires C, 68·4; H, 13·9; N, 17·7%; equiv., 79).

The *dihydrochloride*, prepared in dry ether and crystallised from amyl alcohol, had m. p. 250° (decomp.) (Found: Cl, 30·5. $C_9H_{22}N_2,2HCl$ requires 30·7%). The *dibenzoyl* derivative was prepared by the Schotten–Baumann method; crystals from alcohol, m. p. 133° (Found: C, 75·2; H, 8·25; N, 7·8. $C_{23}H_{30}O_2N_2$ requires C, 75·4; H, 8·2; N, 7·6%).

Sufficient chloro-2 : 4-dinitrobenzene was used with sodium acetate in alcohol to substitute both amino-groups, but only one reacted. The 2 : 4-*dinitrophenyl* derivative crystallised from alcohol in orange needles, m. p. 91° (Found: C, 55·6; H, 7·4; N, 17·7. $C_{15}H_{24}O_4N_4$ requires C, 55·6; H, 7·4; N, 17·3%).

2 : 4 : 4 : 6-*Tetramethyl*-2-(2-*amino*isobutyl)-2 : 3 : 4 : 5-*tetrahydropyrimidine* (X).—Mesityl oxide (120 g.) and liquid ammonia (49 g.) were cooled in an autoclave to — 40° and then stirred for 24 hours at room temperature. The autoclave was then cooled again and a further quantity of mesityl oxide (120 g.) and ammonia (26 g.) added. After 6 days at room temperature, the product was removed and cooled to — 40°. The white crystalline solid that separated (58 g.) crystallised from ether in white flakes, m. p. 61—62° [Found: C, 68·0; H, 11·5; N, 19·9; equiv., 71; *M* (in nitrobenzene), 218. $C_{12}H_{25}N_3$ requires C, 68·2; H, 11·8; N, 19·9%; equiv., 70·3; *M*, 211].

This *compound* (X) reacts rapidly with a cold solution of oxalic acid in alcohol, a precipitate of ammonium hydrogen oxalate appearing shortly after the reagents are mixed. From (X) (2·11 g.) and oxalic acid hydrate (3·78 g.) in alcohol (10 ml.) after ½ hour at the b. p., filtration gave ammonium hydrogen oxalate (1·8 g.) and concentration of the filtrate diacetonamine hydrogen oxalate (0·87 g.).

The latter melted at 124—125° after crystallisation (Found: C, 43·0; H, 7·8. Calc. for $C_8H_{17}O_6N$: C, 43·0; H, 7·6%). There were 2·1 g. of uncrystallisable residues.

2 : 4 : 4 : 6-*Tetramethyl*-2-(2-*amino*iso*butyl*)*hexahydropyrimidine*.—Reduction of (X) (21 g.) in alcohol (100 ml.) with sodium (10 g.) followed by the addition of water (50 ml.) gave the *product* as an oil, which was dried and fractionally distilled (yield, 12—14 g.). It was a colourless liquid, b. p. 122—125°/10 mm.; $d_{25°}^{25°}$ 0·8668; $n_D^{25°}$ 1·458 (Found: C, 67·4; H, 13·0; N, 20·1; equiv., 72. $C_{12}H_{27}N_3$ requires C, 67·6; H, 12·7; N, 19·7%; equiv., 71).

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