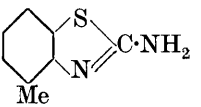
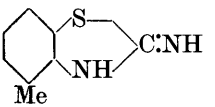
CCCXCVI.—*Aminobenzthiazoles. Part X. The Mobility of the 1-Amino-3-methylbenzthiazole System.*

By Robert Fergus Hunter and Eric Rofe Styles.

In Part III (J., 1926, 1385) it was shown that rational syntheses of 1-aminobenzthiazole and of 1-imino-1 : 2-dihydrobenzthiazole led to a single individual which gave almost quantitatively the 1-imino-2-alkyl-1 : 2-dihydro-derivative on alkylation and 1-acetamidobenzthiazole on acetylation. On the basis of those experiments, the mobility of the symmetrical triad system in 1-aminobenzthiazole was regarded as being established.

That experimental evidence, however, is somewhat slender to carry the superstructure of the theories which have been suggested in Parts V to IX. Moreover, the criticism has still to be met, that the isolation of the second alkyl derivative of 1-aminobenzthiazole was left doubtful; for Pyman (J., 1923, **123**, 367, 3359) has shown that mobile open-chain amidines invariably react in two tautomeric forms on methylation. It was therefore decided to establish the mobility of a typical substituted 1-aminobenzthiazole by means of evidence of the symmetry type (Ingold and Piggott, J., 1922, **121**, 2381) and of the substitution type (*idem*, J., 1923, **123**, 1470) about which there could be no question. The example chosen was 1-amino-3-methylbenzthiazole (I ⇌ II).

(I.)  ⇌  (II.)

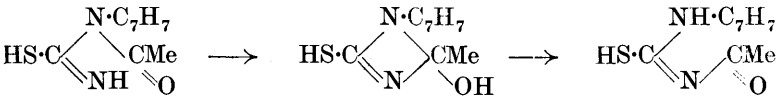
The symmetry test of mobility was first applied. By the rational syntheses mentioned below, 1-*acetamido*-3-*methylbenzthiazole* (III) and

1-*imino*-2-*acetyl*-3-*methyl*-1 : 2-*dihydrobenzthiazole* (IV) were prepared, and these yielded the same tautomeric base (I $\rightleftharpoons$ II) on hydrolysis. Moreover, (III) was obtained in almost quantitative yield by direct acetylation of 1-amino-3-methylbenzthiazole with acetic anhydride.

(III.) $C_6H_3Me\langle\overset{S}{N}\rangle C\cdot NHAc$ $\qquad$ $C_6H_3Me\langle\overset{S}{NAc}\rangle C{:}NH$ (IV.)

Under the usual conditions, bromination of stable acetyl-*o*-tolylthiocarbamide, $C_7H_7\cdot NH\cdot CS\cdot NHAc$ (Hugershoff, *Ber.*, 1899, **32**, 3649; Wheeler, *Amer. Chem. J.*, 1902, **27**, 270) readily produced a *tetrabromide* of 1-acetamido-3-methylbenzthiazole, which gave the expected base (III) on reduction with sulphurous acid. Bromination of labile acetyl-*o*-tolylthiocarbamide, $C_7H_7\cdot NAc\cdot CS\cdot NH_2$, under carefully controlled conditions led to the production of a *tribromide* (compare Hunter, J., 1926, 1395), which gave the isomeric iminoacetyldihydrobenzthiazole (IV) on reduction.

When the temperature was allowed to rise, or when a considerable excess of bromine was employed, during the bromination of *labile* acetyl-*o*-tolylthiocarbamide, the product consisted, not of the expected iminoacetyldihydrobenzthiazole tribromide, but of the tetrabromo-addition compound obtainable by bromination of the isomeric *stable* acetyl-*o*-tolylthiocarbamide. This result was evidently due to conversion of the labile into the stable acetylthiocarbamide under the influence of bromine, similar to the isomerisation which these compounds are known to undergo under the influence of alkalis (Wheeler, *loc. cit.*), followed by thiazole ring formation in the usual way (Hunter and Soyka, J., 1926, 2958). This curious isomerisation may be explained by assuming the formation of an intermediate, unstable, four-membered, heterocyclic compound of the 1 : 3-dimethindiazine type obtained by Ingold and Piggott (J., 1922, **121**, 2797) by the additive union of suitable azomethines (compare also Ingold and Piggott, J., 1923, **123**, 2746; Ingold, J., 1925, **127**, 1141):



The substitution test completely supported the symmetry test of mobility (compare Ingold and Piggott, J., 1923, **123**, 1471), for methylation of the single individual (I $\rightleftharpoons$ II) gave rise to a mixture of 1-*methylamino*-3-*methylbenzthiazole* (V) and 1-*imino*-2 : 3-*dimethyl*-1 : 2-*dihydrobenzthiazole* (VI) containing about 1% of the former. (V and VI are III and IV, respectively, with Me in place of Ac.)

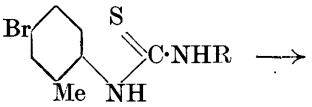
The addition of methyl iodide to mobile and to static amidines (Burtles and Pyman, J., 1923, **123**, 362; Pyman, *ibid.*, pp. 367, 3359) appears to indicate that the first action of methyl iodide on the former is one of direct addition to the double-bonded nitrogen atoms of both phases of the tautomeric system, with the production of isomeric methiodides which pass into the hydriodides of the methyl derivatives derived from the isomeric forms of the mobile individual :

$$\begin{array}{cccccccc} -[H]N{\cdot}C{:}N & & -[H]N{\cdot}C{:}N(MeI) & & -N{:}C{\cdot}N[H](MeI) & & -N{:}C{\cdot}NMe(HI) \\ \uparrow\downarrow & \longrightarrow & + & \longrightarrow & + & \longrightarrow & + \\ -N{:}C{\cdot}N[H] & & -(MeI)N{:}C{\cdot}N[H] & & -(MeI)N[H]{\cdot}C{:}N & & -(HI)NMe{\cdot}C{:}N \end{array}$$

The suggestion (Hunter, J., 1926, 1389) that iminomethyldihydrobenzthiazole is obtained from the iminodihydro-phase by direct replacement of the mobile hydrogen atom (compare von Pechmann, *Ber.*, 1897, **30**, 481; Lander, J., 1903, **83**, 320; Cohen and Marshall, J., 1910, **97**, 329) must therefore be modified (compare Young and Crookes, J., 1906, **89**, 59; Tschitschibabin and Konowalowa, *Ber.*, 1921, **54**, 814). The methylation of semi-cyclic amidines in which one of the nitrogen atoms of the triad system is part of an aromatic heterocyclic nucleus, as in aminothiazoles and α-aminopyridines, is evidently dependent on the aromatic conjugation of the system, a topic which will be more fully discussed in a future paper.

The bromo-addition compounds of 1-amino-3-methylbenzthiazole and its derivatives were reduced by hydriodic acid in acetic acid, but owing to the readiness with which they changed into bromo-substitution compounds it was impossible to estimate the labile bromine iodometrically.

The bromo-substitution compounds were shown to be 5-*bromo*-1-*alkylamino*-3-*methylbenzthiazoles* (VIII) by rational syntheses of the 1-amino-, 1-*n*-propylamino-, and 1-*n*-heptylamino-compounds from the corresponding *p*-bromo-*o*-tolylthiocarbamides (VII).

(VII.)  (VIII.)

EXPERIMENTAL.

(Tautomeric substances are named on the basis of the amino-formulæ.)

Synthesis of 1-*Amino*-3-*methylbenzthiazole from* 1-*Imino*-2-*acetyl*-3-*methyl*-1 : 2-*dihydrobenzthiazole* (IV).—A solution of 5·5 g. of labile acetyl-*o*-tolylthiocarbamide (m. p. 140°) in 50 c.c. of chloro-

form was gradually treated at 0° with bromine (2·5 c.c.) in chloroform (7 c.c.). The liquid was rapidly filtered and kept for 10—15 minutes at about 0° in a vacuum. The yellow needles of the *tribromide* produced, m. p. 173° (decomp.), after drying in a vacuum (Found : Br, 53·5. $C_{10}H_{10}ON_2Br_2S$,HBr requires Br, 53·8%), were suspended in sulphurous acid and treated with sulphur dioxide and thereafter with ammonia (*d* 0·880). The 1-*imino*-2-*acetyl*-3-*methyl*-1 : 2-*dihydrobenzthiazole* obtained crystallised from absolute methyl alcohol in pale yellow prisms, m. p. 170° (Found : N, 13·4; S, 15·5. $C_{10}H_{10}ON_2S$ requires N, 13·6; S, 15·5%). It was refluxed with 25% hydrochloric acid for 24 hours, and the cooled solution neutralised with ammonia. The 1-amino-3-methylbenzthiazole produced crystallised from alcohol in small white plates which, alone or mixed with a genuine specimen, melted at 136° (Found : S, 19·7. Calc. : S, 19·5%).

Synthesis from 1-*Acetamido*-3-*methylbenzthiazole*.—A suspension of stable acetyl-*o*-tolylthiocarbamide (m. p. 187°) (2 g.) in chloroform (15 c.c.) was slowly treated with bromine (2 c.c. in 3 c.c. of chloroform), and the resulting solution refluxed for 10 minutes. On cooling, a *tetrabromide* separated in orange needles, which were dried in a vacuum; m. p. 140° (decomp.) (Found : Br, 61·1. $C_{10}H_{10}ON_2Br_4S$ requires Br, 60·8%). 1-*Acetamido*-3-*methylbenzthiazole*, obtained from this by reduction with sulphurous acid, separated from ethyl acetate in lustrous needles, m. p. 258° (Found : N, 13·8; S, 15·7. $C_{10}H_{10}ON_2S$ requires N, 13·6; S, 15·5%); it was identical with the acetyl derivative obtained by boiling a solution of 1-amino-3-methylbenzthiazole in acetic anhydride for a few minutes. On hydrolysis with 25% hydrochloric acid, 1-amino-3-methylbenzthiazole was obtained and identified in the usual way.

Bromination of 1-acetamido-3-methylbenzthiazole (0·5 g. in 25 c.c. of chloroform; 0·6 c.c. of bromine) yielded an unstable *hexabromide*, which crystallised in glistening orange plates, m. p. 255—258° (decomp.; loss of bromine at about 130°) (Found : Br, 69·8. $C_{10}H_{10}ON_2Br_6S$ requires Br, 70·0%).

Synthesis from 1-*Amino*-3-*methylbenzthiazole Dibromide Hydrobromide*.—A suspension of 2 g. of *o*-tolylthiocarbamide in 16 c.c. of chloroform was treated with bromine (2 c.c. in 3 c.c. of chloroform); the thiocarbamide dissolved and a yellow precipitate separated and redissolved with evolution of heat and hydrogen bromide, the solution boiling spontaneously. After a few minutes' refluxing, the *dibromide hydrobromide* crystallised in large, lustrous, orange-yellow plates, m. p. 129° (decomp. with efferv.) [Found : Br, 59·9; Br (labile), 35·0. $C_8H_8N_2Br_2S$,HBr requires Br, 59·3; Br (labile), 39·5%]. The low titration value is due to a certain

amount of the bromo-addition compound passing into 5-bromo-1-amino-3-methylbenzthiazole hydrobromide, a change which has been observed by Prof. R. W. West and his collaborators (private communication) in the case of perbromides of aromatic bases having a free *para* position (compare also Fries, *Annalen*, 1906, **346**, 128). The bromide dissolved in 80% alcohol, from which *5-bromo-1-amino-3-methylbenzthiazole hydrobromide* crystallised in small white needles, m. p. 280—290° (with charring) (Found : Br, 49·1. $C_8H_7N_2BrS,HBr$ requires Br, 49·4%). Treatment of the bromide with sulphurous acid and subsequent basification, however, gave a quantitative yield of 1-amino-3-methylbenzthiazole, m. p. 136°.

When prepared as above, in large crystals, the dibromide hydrobromide can be preserved in a sealed vessel for more than a year. Small crystals (having the same crystalline structure and m. p. as before), obtained by alteration in the conditions of concentration and separation, are sensitive to moisture; one specimen, on exposure to the laboratory atmosphere for 30 hours, was completely converted into *1-amino-3-methylbenzthiazole hydrobromide* (m. p. 220°), identical with the hydrobromide prepared by concentrating a solution of 1-amino-3-methylbenzthiazole in 20% hydrobromic acid; this crystallised in small needles, m. p. 220—222° (Found : Br, 32·0. $C_8H_8N_2S,HBr$ requires Br, 32·6%).

1-Amino-3-methylbenzthiazole Tetrabromide.—When a solution of 0·6 g. of 1-amino-3-methylbenzthiazole in 10 c.c. of chloroform was gradually treated with a solution of bromine (0·4 c.c. in 5 c.c. of chloroform) a *tetrabromide* was obtained in yellowish-brown prisms, m. p. 302° (loss of colour at 130—140°) (Found : Br, 65·9. $C_8H_8N_2Br_4S$ requires Br, 66·1%).

Methylation of 1-Amino-3-methylbenzthiazole and the Isolation of 1-Methylamino-3-methylbenzthiazole and of 1-Imino-2 : 3-dimethyl-1 : 2-dihydrobenzthiazole.—A mixture of 1-amino-3-methylbenzthiazole (3·5 g.) and methyl iodide (2·5 c.c.) was heated at 100° for 24 hours; the product was basified with ammonia and extracted with ether. The clear oil obtained after removal of the ether solidified on keeping (sometimes with considerable difficulty) and was fractionally crystallised from methyl alcohol. The first crop of crystals (about 0·02 g.) had m. p. 126°, which rose to 128° on recrystallisation and was unaltered by admixture with a genuine specimen of 1-methylamino-3-methylbenzthiazole prepared from *s-o*-tolylmethylthiocarbamide. (A mixture of 1-methylamino-3-methylbenzthiazole and 1-amino-3-methylbenzthiazole melted at 103—105° after softening at about 100°.) The more soluble crops were dissolved in dilute methyl alcohol and the solution was concentrated at 16°/20 mm.; slightly impure 1-imino-2 : 3-dimethyl-

1 : 2-dihydrobenzthiazole then separated. This product had m. p. 83—84°, and m. p. 84—85° when mixed with a genuine specimen of 1-imino-2 : 3-dimethyl-1 : 2-dihydrobenzthiazole prepared from *as*-*o*-tolylmethylthiocarbamide; on recrystallisation the m. p. rose to 85—86°. The crude iminomethyl base was acetylated with acetic anhydride, and the product fractionally crystallised from alcohol; the bulk of the acetyl derivative consisted of the 1-acetimido-compound, which had m. p. 143°, and m. p. 145° when mixed with a genuine specimen of 1-acetimido-2 : 3-dimethyl-1 : 2-dihydrobenzthiazole. The mother-liquor from the acetylation, however, yielded a high-melting fraction, which, after recrystallisation, had m. p. 258° alone and when mixed with 1-acetamido-3-methylbenzthiazole.

Synthesis of 1-*Methylamino*-3-*methylbenzthiazole and of* 1-*Imino*-2 : 3-*dimethyl*-1 : 2-*dihydrobenzthiazole from the Corresponding Tolylmethylthiocarbamides.*—*o*-Tolylthiocarbimide was conveniently prepared in 80—90% yield (b. p. 238—240°/760 mm.) by refluxing quantities of 50 g. of *s*-di-*o*-tolylthiocarbamide with acetic anhydride (60 c.c.) for 10 minutes, being isolated by distillation in steam. *o*-Tolylthiocarbamide separated from 50% alcohol in glistening white needles, m. p. 160° (Dyson and George, J., 1924, **125**, 1703, recorded m. p. 156°).

s-o-*Tolylmethylthiocarbamide*, prepared by treating 5 g. of *o*-tolylthiocarbimide in 20 c.c. of absolute alcohol with a 30% excess of 33% methylamine in the same solvent, crystallised in glistening prisms, m. p. 161° (Found : S, 17·8. $C_9H_{12}N_2S$ requires S, 17·8%).

Bromination. A suspension of 2 g. of *s*-*o*-tolylmethylthiocarbamide in 10 c.c. of chloroform was treated with bromine (2 c.c. in 3 c.c. of chloroform) and the liquid was refluxed vigorously for 10 minutes and then concentrated in a vacuum at laboratory temperature; a *tetrabromide* crystallised in glistening red needles, m. p. 75° (decomp.) (Found : Br, 64·4. $C_9H_{10}N_2Br_4S$ requires Br, 64·2%). This compound was extremely sensitive to moist air and became yellow with loss of bromine on being exposed to the laboratory atmosphere for a few minutes.

When the bromination was carried out as above, but instead of being vigorously boiled, the solution was gently warmed on the steam-bath, a *tribromide* was obtained on concentration in a vacuum, which was probably the dibromide of a hydrobromide analogous to the compound obtained from *o*-tolylthiocarbamide itself. It formed glistening yellow prisms, m. p. 113° (Found : Br, 57·6. $C_9H_{10}N_2Br_2S,HBr$ requires Br, 57·3%). Attempts to determine its molecular weight in boiling chloroform (previously dried) were unsuccessful owing to dissociation with the liberation of free

bromine; on prolonged boiling in chloroform, hydrogen bromide also was liberated.

Both the tetrabromide and the tribromide dissolved in sulphurous acid, and on basification with ammonia, *1-methylamino-3-methylbenzthiazole* separated, which crystallised from alcohol in lustrous needles, m. p. 130° (Found : N, 15·9; S, 18·2. $C_9H_{10}N_2S$ requires N, 15·7; S, 18·0%). The *acetyl* derivative, prepared as in the case of the isomeric iminodihydro-derivative, crystallised from alcohol–ethyl acetate in white needles, m. p. 133° (Found : S, 14·6. $C_{11}H_{12}ON_2S$ requires S, 14·5%).

as-o-*Tolylmethylthiocarbamide*.—Addition of methyl-*o*-toluidine in chloroform to thiocarbonyl chloride in water (compare Dyson and George, *loc. cit.*) gave a thiocarbamyl chloride which yielded uncrystallisable oils on treatment with ammonia. The thiocarbamide was therefore prepared by way of the thiocyanate. A solution of 10 g. of methyl-*o*-toluidine hydrochloride and 7 g. of potassium thiocyanate in 50 c.c. of water was heated for 3 hours on a steam-bath and then extracted with chloroform. The gum obtained by evaporation of the chloroform crystallised from methyl alcohol in small, white needles (3·5 g.), m. p. 107—108° (Found : S, 17·6%).

Bromination. The solution obtained from 1 g. of the thiocarbamide, 7 c.c. of chloroform, and 1 c.c. of bromine was refluxed for 3 minutes and then concentrated under reduced pressure at laboratory temperature. The orange bromo-addition compound obtained was dissolved in sulphurous acid and the filtered solution was basified and extracted with chloroform. The gum obtained by evaporation of the chloroform was dissolved in 80—90% methyl alcohol, and the solution concentrated at 16°/20 mm.; *1-imino-2 : 3-dimethyl-1 : 2-dihydrobenzthiazole* crystallised in small white plates, m. p. 86° (Found : S, 18·1%). The *acetyl* derivative separated from alcohol in glistening plates, m. p. 147° (Found : S, 14·7%).

5-Bromo-1-amino-3-methylbenzthiazole.—A suspension of 0·9 g. of 5-bromo-*o*-tolylthiocarbamide (m. p. 194°. Dyson and George, J., 1924, **125**, 1706, give 186°) in chloroform (10 c.c.) was treated with bromine (1·1 c.c. in 1 c.c. of chloroform), and the solution refluxed for 10 minutes; a *dibromide hydrobromide* crystallised in orange, glistening prisms, which turned white at 130—140° but were unmelted at 250° (Found : Br, 66·3. $C_8H_7N_2Br_3S,HBr$ requires Br, 66·1%). On treatment with sulphurous acid and ammonia, it yielded 5-bromo-1-amino-3-methylbenzthiazole, which crystallised from alcohol in needles, m. p. 212°, and was identical with the base liberated from the hydrobromide obtained by the action of alcohol on 1-amino-3-methylbenzthiazole dibromide hydrobromide (Found : Br, 32·7. Calc. : Br, 32·9%).

s-o-*Tolyl*-n-*propylthiocarbamide* was prepared by warming a solution of *o*-tolylthiocarbimide in alcohol with 30% excess of *n*-propylamine. It separated from the solution after concentration and crystallised from alcohol in colourless prisms, m. p. 66° (Found : S, 15·5. $C_{11}H_{16}N_2S$ requires S, 15·4%).

1-n-*Propylamino*-3-*methylbenzthiazole Tetrabromide*.—A solution of 2 g. of *s*-*o*-tolyl-*n*-propylthiocarbamide in 10 c.c. of chloroform was treated with bromine (2 c.c. in 3 c.c. of chloroform), and the liquid vigorously refluxed for 10 minutes. On concentration in a vacuum the *tetrabromide* crystallised in brilliant red needles, m. p. 71° (efferv. at 130°) (Found : Br, 60·6. $C_{11}H_{14}N_2Br_4S$ requires Br, 60·8%). On treatment with sulphurous acid followed by ammonia the *base* was obtained; it crystallised from methyl alcohol in colourless needles, m. p. 62° (Found : S, 15·7. $C_{11}H_{14}N_2S$ requires S, 15·5%). The *hydrobromide* crystallised from ethyl acetate in prisms, m. p. 179° (decomp. after previous sintering) (Found : N, 10·0. $C_{11}H_{14}N_2S$,HBr requires N, 9·8%).

The *acetyl* derivative formed colourless prisms, m. p. 61° (Found : S, 13·0. $C_{13}H_{16}ON_2S$ requires S, 12·9%).

When a solution of the tetrabromide in alcohol was diluted with a small quantity of water and concentrated, the *hydrobromide* of the bromo-substitution derivative separated; this crystallised from alcohol in tufts of fine white needles, m. p. 259° (Found : Br, 45·0. $C_{11}H_{13}N_2BrS$,HBr requires Br, 43·7%).

s-5-*Bromo*-o-*tolyl*-n-*propylthiocarbamide*, prepared from 5-bromo-*o*-tolylthiocarbimide and *n*-propylamine, crystallised from methyl alcohol in slender white needles, m. p. 79° (Found : S, 11·4. $C_{11}H_{15}N_2BrS$ requires S, 11·2%).

5-*Bromo*-1-n-*propylamino*-3-*methylbenzthiazole*.—A solution of 1 g. of the bromotolylthiocarbamide in chloroform (10 c.c.) was refluxed with 1·2 c.c. of bromine for 15 minutes, cooled, and concentrated in a vacuum; an ill-defined bromo-addition compound separated (m. p. about 140°), which was reduced with sulphurous acid in the usual way. On recrystallisation from methyl alcohol, 5-*bromo*-1-n-*propylamino*-3-*methylbenzthiazole* was obtained, m. p. 82°, which was identical with the base (m. p. 82°) liberated from the last-mentioned hydrobromide (Found : Br, 28·3. $C_{11}H_{13}N_2BrS$ requires Br, 28·1%).

s-o-*Tolyl*-n-*heptylthiocarbamide* was prepared from *o*-tolylthiocarbimide and *n*-heptylamine and obtained as a viscous oil which gradually solidified in a vacuum. It crystallised from alcohol in large, colourless prisms, m. p. 98° (Found : S, 12·3. $C_{15}H_{24}N_2S$ requires S, 12·1%).

Bromination. When 2 g. of the heptylthiocarbamide were

brominated as in the case of the propyl compound, an unstable *hexabromide* was obtained in orange-red needles, m. p. 53° (sintering at 45°) (Found : Br, 64·4. $C_{15}H_{22}N_2Br_6S$ requires Br, 64·7%).

1-n-*Heptylamino*-3-*methylbenzthiazole* crystallised with difficulty from dilute methyl alcohol in white prisms, m. p. 57° (Found : S, 12·4. $C_{15}H_{22}N_2S$ requires S, 12·2%). The *acetyl* derivative formed stellate aggregates of needles, m. p. 73° (Found : S, 10·6. $C_{17}H_{24}ON_2S$ requires S, 10·5%).

The hexabromide dissolved in aqueous alcohol, giving the *hydrobromide* of the bromo-substitution derivative, which crystallised from alcohol in small, white plates, m. p. 220° (Found : Br, 37·6. $C_{15}H_{21}N_2BrS,HBr$ requires Br, 37·9%).

s-5-*Bromo*-o-*tolyl*-n-*heptylthiocarbamide* separated from dilute methyl alcohol in small white needles, m. p. 71° (Found : S, 10·1. $C_{15}H_{23}N_2BrS$ requires S, 9·3%).

5-*Bromo*-1-n-*heptylamino*-3-*methylbenzthiazole*.—The bromo-addition compound obtained from the bromotolylthiocarbamide was reduced and the 5-*bromo-base* produced was crystallised from dilute methyl alcohol; it had m. p. 75° alone and when mixed with the base (m. p. 75°) liberated from the preceding hydrobromide (Found : N, 8·6; Br, 23·7. $C_{15}H_{21}N_2BrS$ requires N, 8·2; Br, 23·5%).

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