Aspects of Mass Spectra of Organic Compounds. Part XI.[1] Rearrangements in Benzoylhydrazines

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The mass spectra of N'-substituted N-benzoylhydrazines show abundant molecular ions and do not show skeletally rearranged ions. However, the diacylated hydrazine, NN'-dibenzoylphenylhydrazine, shows an unusual loss of 122 a.m.u. from the molecular ion, which involves skeletal rearrangement and loss of the elements of benzoic acid. A short study of the substituent effect on this rearrangement shows the complex competition between simple cleavage and rearrangement processes. Rearrangement of both aroyl groups is observed in some cases, which can be explained by formation of an intermediate imide, followed by 1,3-aroyl migration from nitrogen to oxygen and/or by a five-membered cyclic transition state. The mass spectra of other hydrazine derivatives showing possible 1,3-rearrangements from nitrogen to oxygen are reported, supplementing our earlier work. Some erroneous reports in the literature, where elemental compositions were incorrectly assumed, are corrected by determining them from high resolution data.

DURING our studies of the skeletal rearrangements in the mass spectra of amides and related compounds,[1,2] we observed an unusual loss of 122 a.m.u. (the elements of benzoic acid) from the molecular ion of NN'-dibenzoylphenylhydrazine (1).[3] We then examined the mass spectra of other acylated hydrazines, but the work remained unpublished. A recent publication by Porter and Seif[4] prompts us to publish this work because our results differ significantly from those of these authors. On the basis of some ambiguous deuterium-labelling studies and without the benefit of a high resolution measurement, Porter and Seif[4] recently incorrectly reported that the loss of 122 a.m.u. involves a concerted loss of benzamide and an additional hydrogen atom. We report here the mass spectra of a variety of benzoylhydrazines to supplement and, where necessary, to correct the independent work published recently.[4,5] We also report a study of substituent effects on the unusual rearrangement in the mass spectrum of the hydrazine derivative (1).

RESULTS AND DISCUSSION

Low resolution mass spectra of a series of mono-benzoylhydrazines (2)—(7) and some dibenzoylhydrazines (8)—(11) are shown in Table 1. The spectra of various derivatives of dibenzoylphenylhydrazine (1) are shown in Tables 2 and 3. Other miscellaneous results are shown in Table 4.

In all cases the molecular ions of the monobenzoylhydrazines (2)—(7) are relatively abundant and skeletally rearranged ions are not apparent (Table 1). N-Benzoyl-N'-methylhydrazine (3) fragments by pathways involving hydrogen transfers in the losses of CH_2N and CH_3N from the molecular ion. A hydrogen atom from the methyl group could be transferred to the oxygen atom in a process formally analogous to the McLafferty re-

arrangement of ketones. A similar process has been suggested to explain the mass spectrum of NN-dibenzoyl-$N'N'$-dimethylhydrazine.[5]

TABLE 1

Normalised mass spectra of mono- and di-benzoyl-hydrazines (2)—(11)

	RNH·NHBz	m/e (%)
(2)	R = H (120°)[a]	51 (16), 77 (65), 105 (100), 106 (8), 136 (20)
(3)	R = Me (200°)[b]	45 (10), 50 (8), 51 (16), 76 (5), 77 (52), 78 (6), 103 (9, C_7H_5N), 105 (100, C_7H_5O), 106 (8), 121 (14, C_7H_7NO), 122 (14, C_7H_8NO), 150 (3, $C_8H_{10}N_2O$)
(4)	R = CH_2Ph (200°)	51 (17), 65 (8), 77 (50), 91 (46), 105 (100), 106 (83, C_7H_8N), 122 (30, C_7H_8NO), 149 (6, $C_8H_9N_2O$), 226 (6, $C_{14}H_{14}N_2O$)
(5)	R = Ph (180°)	51 (10), 77 (40), 105 (100), 106 (8), 107 (11), 212 (40), 213 (7)
(6)	R = 2-pyridyl (200°)	51 (11), 77 (45), 78 (8), 79 (7), 105 (100), 106 (9), 108 (32, $C_6H_6N_3$), 195 (5, $C_{12}H_9N_3$), 196 (6, $C_{12}H_{10}N_3$), 213 (32, $C_{12}H_{11}N_3O$)
(7)	MeNBz·NH₂ (200°)	51 (20), 77 (65), 78 (5), 105 (100), 106 (6), 150 (24)
	$R_ANBz·NR_BBz$	
(8)	$R_A = R_B = H$ (180°)	51 (6), 77 (40), 105 (100), 106 (8), 241 (15)
(9)	$R_A = Me, R_B = H$	51 (8), 77 (34), 105 (100), 106 (10), 122 (7, C_7H_8NO), 149 (3, $C_8H_9N_2O$), 226 (2, $C_{14}H_{12}NO_2$), 254 (6, $C_{15}H_{14}N_2O_2$)
(10)	$R_A = R_B = Me$ (200°)	51 (5), 77 (31), 105 (100), 106 (9), 119 (<1), 134 (2), 135 (4), 136 (7), 147 (2), 163 (6), 240 (1), 268 (<0.5)
(11)	$R_A = CH_2Ph$, (200°)[e] $R_B = H$	51 (7), 77 (30), 91 (8), 105 (100), 106 (9), 122 (3, C_7H_8NO), 209 (10, $C_{14}H_{11}NO$), 210 (10, $C_{14}H_{12}NO$), 211 (2), 225 (2, $C_{14}H_{13}N_2O$), 226 (2, $C_{14}H_{12}NO_2$), 314 (<0.5, $C_{21}H_{18}N_2O$), 330 (<0.5)

[a] Source temp. (°C). [b] Heated inlet system at 200°. [e] MS902.

The mass spectrum of the benzylhydrazine derivative (4) shows a peak at m/e 122, C_7H_8NO, due to double

[1] Part X, T. W. Bentley and R. A. W. Johnstone, *J. Chem. Soc. (B)*, 1971, 1804.

[2] T. W. Bentley, R. A. W. Johnstone, and D. W. Payling, *Chem. Comm.*, 1968, 1154.

[3] R. A. W. Johnstone and D. W. Payling, unpublished results.

[4] Q. N. Porter and A. E. Seif, *Austral. J. Chem.*, 1972, **25**, 523.

[5] Q. N. Porter and A. E. Seif, *Org. Mass Spectrometry*, 1970, **4**, 361.

TABLE 2

Normalised mass spectra of deuteriated derivatives
(12)—(15) of NN'-dibenzoylphenylhydrazine

PhNCOR^1·NR^{3}COR^2 m/e (%)

(1)   R^1 = R^2 = Ph   77 (25), 105 (100), 106 (8), 194 (4),
      R^3 = H (200°)   316 (4)

(1) [a]   77 (29), 105 (100), 106 (10), 194 (4·1),
                                   316 (3·9)

(12) [a] R^1 = R^2 = C$_6$D$_5$,   82 (29), 110 (100), 111 (10), 199 (4·1),
          R^3 = H   326 (4·6)

(13) [a] R^1 = R^2 = C$_6$D$_5$,   82 (30), 110 (100), 111 (10), 199 (3·7),
          R^3 = D   327 (4·7)

(14)   R^1 = R^2 = C$_6$H$_5$,   77 (25), 105 (100), 106 (8), 194 (3),
       R^3 = D (180°)   317 (4)

(15)   R^1 = C$_6$D$_5$,   77 (32), 78 (0·7), 79 (0·08), 80 (0·04),
       R^2 = Ph, R^3 = H   81 (10), 82 (21), 105 (65), 106 (1·2),
       (180°)   107 (1·0), 108 (1·2), 109 (42), 110
                                   (100), 194 (1·2), 195 (0·2), 196 (0·04),
                                   197 (0·08), 198 (0·25), 199 (0·52),
                                   316 (0·08), 317 (0·04), 318 (0·03),
                                   320 (0·4), 321 (0·9)

[a] Results from ref. 4.

TABLE 3

Substituent effects on the mass spectra of diaroylphenyl-
hydrazines (16)—(22)

PhN(OC·C$_6$H$_4$Y-p)·-
NH(OC·C$_6$H$_4$Z-p)   (m/e (%))

(1)   Y = Z = H   77 (25), 105 (100), 106 (8), 194 (4,
      (200°)   C$_{13}$H$_{10}$N$_2$), 316 (4, C$_{20}$H$_{16}$N$_2$O$_2$)

(16) Y = Me, Z = H   77 (11), 91 (20), 105 (19), 119 (100),
      (210°)   120 (10), 194 (1·2), 208 (0·75),
                        330 (2·5)

(17) Y = NO$_2$, Z = H   77 (22), 105 (100), 106 (8), 120 (6),
      (210°)   150 (13), 151 (3·5), 194 (4, C$_{13}$H$_{10}$-
                        N$_2$), 211 (2·5, C$_{13}$H$_{11}$N$_2$O), 361 (7)

(18) Y = OMe, Z = H   77 (11), 92 (7), 105 (10), 107 (6),
      (200°)   135 (100), 136 (9), 194 (<0·4,
                        C$_{13}$H$_{10}$N$_2$), 346 (2)

(19) Y = Z = OMe   77 (8), 92 (5), 107 (4), 135 (100),
      (200°)   136 (10), 224 (1), 376 (5)

(20) Y = H, Z = OMe   77 (35), 92 (7), 105 (100), 106 (8),
      (200°)   107 (6), 135 (90), 136 (9), 194 (<0·4),
                        224 (4), 346 (11)

(21) Y = H, Z = NO$_2$ 77 (30), 105 (100), 106 (9), 120 (1),
      (200°)   150 (3), 361 (2)

(22) Y = H, Z = Me   77 (23), 91 (15), 105 (100), 106 (8),
      (210°) [a]   119 (49), 120 (4), 194 (2), 208 (4),
                        330 (4)

[a] MS902.

TABLE 4

Normalised mass spectra

m/e (%)

(23) (PhNBz)$_2$ 51 (3), 77 (25), 105 (100), 106 (7), 197 (1,
     (210°) [a]   C$_{13}$H$_{11}$NO), 287 (3, C$_{19}$H$_{15}$N$_2$O), 299 (1,
                      C$_{20}$H$_{15}$N$_2$O), 392 (10, C$_{26}$H$_{20}$N$_2$O$_2$)

(24) PhNH·NPhBz   51 (10), 77 (68), 78 (6), 93 (6), 105 (60),
     (210°) [a]   106 (5), 183 (100), 184 (15), 288 (20),
                    289 (4)

(25) MeNBz·NBz$_2$ 51 (8), 77 (44), 105 (100), 106 (9), 237 (34,
     (130°)   C$_{15}$H$_{13}$N$_2$), 238 (6), 358 (3)

[a] MS902.

hydrogen transfer and a peak at m/e 106 corresponding
to cleavage of the N–N bond. Cleavage of the N–N
bond is also apparent in the peaks at m/e 107 in the

spectrum of the phenylhydrazine derivative (5) and
m/e 108 in that of the 2-pyridylhydrazine derivative (6).

The 2-pyridylhydrazine derivative (6) fragments to
give ions at m/e 195 and 196 corresponding to losses of
H$_2$O and HO from the molecular ion. A metastable ion
was observed for the process m/e 213 (M$^+$) —► m/e 196.
This unusual fragmentation was not observed in the
mass spectra of 3- and 4-pyridyl analogues.

The extent to which mass spectrometry can be used to
distinguish between isomeric structures is important
theoretically and in work on structure elucidation by
mass spectrometry. The mass spectrum of N-benzoyl-
N-methylhydrazine (7) differs from that of its isomer (3)
in that it does not show loss of 28 or 29 a.m.u. The
results of Porter and Seif [4,5] show that different hydrogen
transfer processes occur in the mass spectra of the two
isomeric dibenzoyldimethylhydrazines. Thus mass
spectrometry could be useful for structure elucidation in
such cases.

Porter and Seif [4] have already reported and discussed
the mass spectra of the dibenzoylhydrazines (8)—(10),
and our independent high resolution studies substantiate
their results (see Table 1). They also reported the mass
spectrum of the benzylhydrazine derivative (11), which
appeared to be unexceptional. In remarkable contrast,
using the MS12 mass spectrometer, we were unable to
detect a significant molecular ion at m/e 330; the first
significant peak at high mass : charge ratio was at
m/e 314, formally corresponding to loss of an oxygen
atom from the molecular ion. However, the spectrum
obtained on the MS902 instrument (see Table 1) at
several temperatures was similar to the published one,[4]
except that it was difficult to reproduce the region above
m/e 300; very weak peaks at m/e 313, 314, 330, and 331
varied in intensity, and it was sometimes difficult to
pick out the molecular ion. Compared with spectra
obtained with the MS902 instrument, those obtained
with the MS12 had a much more intense (ca. 10%) peak
at m/e 211 and a much weaker peak (<2%) at m/e 209.
Also, the peak at m/e 225 was insignificant in some
spectra from the MS12.

A much less confusing difference between our work
and Porter and Seif's [4] is in the interpretation of the
one-step loss of 122 a.m.u. from the molecular ion of
NN'-dibenzoylphenylhydrazine (1), showing a relatively
abundant metastable ion at m/e 119·1. Table 2 shows
the mass spectra of some deuteriated analogues of
compound (1) [see (12)—(15)]. Porter and Seif studied
compounds (12) and (13) and correctly concluded that
either R^1 or R^2 was ejected along with R^3. In com-
pound (1), R^1 and R^3 would account for only 78 of the
122 a.m.u. that are lost. We have found by high
resolution measurements that the remaining 44 a.m.u.
correspond to the elements of CO$_2$, not CH$_2$NO as
assumed by Porter and Seif.[4] Thus the 122 a.m.u.
corresponds to the elements of benzoic acid and we have
observed a surprising new skeletal rearrangement and
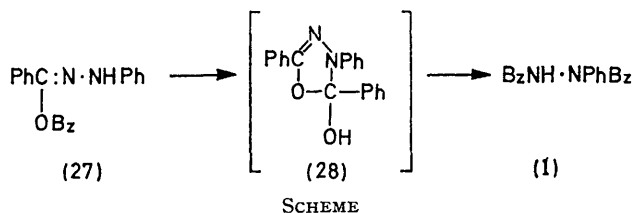not a double hydrogen rearrangement.

Our interest in substituent effects in mass spectro-

metry [6,7] prompted a study of the substituent effects on these fragmentation processes. The mass spectra of the series of compounds we have studied are shown in Table 3. Simple cleavage to form aroyl cations is always very favourable, especially when $Y = OCH_3$. The ease of formation of aroyl cations appears to be in the order: $Y = OCH_3 > Y = CH_3 > Y = H > Z = OCH_3 > Z = CH_3 > Z = H > Y = NO_2 > Z = NO_2$. Formation of R^1CO^+ appears to be intrinsically more favourable than R^2CO^+, presumably because the phenyl group would stabilise the radical on the α-nitrogen atom. Also electron-withdrawing substituents destabilise aroyl cations, so the observed order is readily explained in a qualitative way. Supporting evidence that formation of R^1CO^+ is favoured over R^2CO^+ comes from the mass spectrum of the deuteriated compound (15), where peaks at m/e 109 and 110 from R^1CO are about twice as intense as the peak at m/e 105 due to R^2CO^+. Formation of ions at m/e 77, 81, and 82 probably requires more energy and is less selective.

Ions at m/e 194, 198, and 199 in the mass spectrum of compound (15) show that loss of R^1CO_2H is preferred by about 1·5 : 1 over loss of R^2CO_2H. In the series of substituted compounds (16)—(22) loss of the aryl group containing Y (*i.e.* R^1) is preferred in all cases. A possible intermediate in this unusual skeletal rearrangement is the imide (26), which, by analogy with our extensive

$$\left[PhNBz \cdot NHBz \right]^{+\cdot} \longrightarrow \left[PhN \cdot NHBz_2 \right]^{+\cdot}$$
$$(26)$$
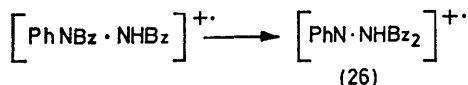
studies of related rearrangements,[1,2] would be expected to undergo a transfer of benzoyl and hydrogen from nitrogen to oxygen. Formation of the imide (26) involving a 1,2-shift of benzoyl from nitrogen to nitrogen, is a new skeletal rearrangement postulated to explain loss of CH_2N from the hydrazine derivative (9).[4] Incidentally the thermal process (27) $\longrightarrow$ (28) (Scheme)

SCHEME

appears to be similar to the reverse of the skeletal rearrangement process for loss of $C_7H_6O_2$.[8] Certainly a direct 1,4-transfer of benzoyl from nitrogen to oxygen could be involved rather than, or in addition to, a 1,2-followed by a 1,3-shift. However, the latter mechanism,

involving the potentially symmetrical intermediate (26), explains the lack of complete specificity in which an aroyl group is lost (Table 3) from the molecular ions of the series of substituted derivatives (16)—(22).

A difficulty with the foregoing speculative interpretation involving a symmetrical intermediate imide (26) is that it does not readily explain why the deuteriated compound (15) (see Table 2) shows about a 1·5 : 1 preference for loss of R^1 rather than R^2. The small difference in ionisation potentials between benzene and [$^2H_6$]benzene,[9] and the small difference (0·01) in pK_a values between benzoic acid and [$^2H_5$]benzoic acid,[10] suggest that the preference can *not* be explained by an isotope effect. Also the mass spectra of isomers, for example compounds (16) and (22), do not show the same relative amounts of rearrangement of each aroyl group; there is a preference for loss of the substituent Y (*i.e.* R^1CO_2H). Thus if the intermediate (26) is formed during the rearrangement, it is probably not symmetrical.

The minor loss of 93 a.m.u. from compound (23) has been assumed to be C_6H_7N,[4] whereas recourse to high resolution measurement or analogy with our earlier work [1,2] would indicate that the group C_6H_5O was lost (see Table 4). The process is formally explained by 1,3-shift of phenyl from nitrogen to oxygen.[1] Simple cleavage processes appear to predominate in the mass spectrum of compound (24), but a similar 1,3-shift of a benzoyl group from nitrogen to oxygen explains the abundant loss of $C_7H_5O_2$ from tribenzoylmethylhydrazine (25). The mass spectra of the foregoing compounds are shown in Table 4.

CONCLUSION

The mass spectra of di- or tri-aroylhydrazines show significant skeletal rearrangement processes, involving rearrangements to (or by) the oxygen atom(s). Although the major peaks in the spectra are due to aroyl cations and derived fragment ions, these do not give specific structural information. The rearrangement processes are important because they give relatively intense metastable ions, and the normal ions formed after rearrangement are of comparable intensity to the molecular ions.

Although deuterium labelling has been described as the ' single most useful ' method for studying fragmentation processes in organic mass spectrometry,[11] high resolution measurements provide a lot of useful and important information quickly. The elemental composition of an ion is the most definite property (apart

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[7] For reviews see M. M. Bursey and M. K. Hoffman in ' Mass Spectrometry, Techniques and Applications,' ed. G. W. A. Milne, Wiley, London, 1971, pp. 387—397; T. W. Bentley and R. A. W. Johnstone, *Adv. Phys. Org. Chem.*, 1970, **8**, 229—235; I. Howe in ' Mass Spectrometry,' Specialist Periodical Report, The Chemical Society, 1971, vol. 1, pp. 66—80.

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[11] R. G. Cooks, I. Howe, and D. H. Williams, *Org. Mass. Spectrometry*, 1969, **2**, 148. This important review also emphasises some of the pitfalls in interpreting the results of deuterium labelling in mass spectrometry.

from m/e) we can measure and should be determined whenever possible.

EXPERIMENTAL

Except where otherwise stated the mass spectra were recorded on a G.E.C.–A.E.I. MS12 spectrometer at 70 eV and at source temperatures indicated in the Tables. Data reported are averages of at least two spectra. Similar spectra were obtained at high resolution, by use of a Ferranti Argus 500 computer data acquisition system and an MS902 spectrometer; in general we found that signals at higher m/e values were relatively less intense than those observed with the MS12.

Compounds (2) (Koch–Light), (3),[12] (4),[13] (5),[14] (6),[15] (8),[16] (9),[17] and (10) [18] were readily available and their characterisation was straightforward. M.p.s were in agreement with literature values, purity was checked by t.l.c. (often on silica gel G, eluted with chloroform) and any doubts about structure were settled by n.m.r. spectroscopy.

aroyl chloride with phenylhydrazine gave N-aroyl-N'-phenylhydrazine, which then reacted with a second mole of acid chloride. The intermediates are well characterised compounds: N-benzoyl-N'-phenylhydrazine (5), m.p. 170—171° (lit.,[14] 170°); N-p-anisoyl-N'-phenylhydrazine, m.p. 177—179° (lit.,[23] 179°); N-phenyl-N'-p-toluoylhydrazine, m.p. 167—168° (lit.,[24] 165—166°); N-p-nitrobenzoyl-N'-phenylhydrazine, m.p. 200—202° (lit.,[25] 200—201°). A typical synthesis of a diaroyl compound is now described.

N-p-*Anisoyl*-N'-*benzoyl*-N-*phenylhydrazine* (18).—A suspension of N-benzoyl-N'-phenylhydrazine (2·2 g) in chloroform (50 ml) was treated with anisoyl chloride (2·3 g), then heated under reflux for 5 min. After cooling, the chloroform layer was stirred with sodium hydrogen carbonate solution overnight. The organic layer was then separated, washed with water, dried ($CaCl_2$), and evaporated. The residue was recrystallised from ethanol (yield 2·3 g, 64%). Compounds (16)—(22) were prepared similarly. Analytical data and m.p.s are given in Table 5.

TABLE 5
Analytical data and m.p.s of diaroylphenylhydrazines

Compound	M.p. (°C)	Formula	Found (%)			Calc. (%)		
			C	H	N	C	H	N
(1) Y = Z = H	177—179 a	$C_{20}H_{16}N_2O_2$	75·6	5·3	8·75	75·9	5·1	8·9
(16) Y = Me, Z = H	157—159	$C_{21}H_{18}N_2O_2$	76·2	5·8	8·7	76·3	5·5	8·5
(17) Y = NO₂, Z = H	167—169	$C_{20}H_{15}N_3O_4$	66·55	4·2	11·6	66·5	4·2	11·6
(18) Y = OMe, Z = H	196—198	$C_{21}H_{18}N_2O_3$	72·7	5·4	8·15	72·8	5·2	8·1
(19) Y = OMe, Z = OMe	199—201	$C_{22}H_{20}N_2O_4$	70·2	5·5	7·6	70·2	5·4	7·4
(20) Y = H, Z = OMe	131—132	$C_{21}H_{18}N_2O_3$	72·8	5·3	8·3	72·8	5·2	8·1
(21) Y = H, Z = NO₂	197—199	$C_{20}H_{15}N_3O_4$	66·3	4·1	11·4	66·5	4·2	11·6
(22) Y = H, Z = Me	160—162	$C_{21}H_{18}N_2O_2$	76·1	5·45	8·5	76·3	5·5	8·5

a Lit.,[14] 178°.

N-*Benzoyl*-N-*methylhydrazine* (7).—Equimolar amounts of methylhydrazine and benzoic anhydride in ether at 0° gave impure material as a yellow oil.[19] The n.m.r. spectrum showed two types of methyl group, the minor one corresponding to the dibenzoyl derivative (9). The oil was dissolved in ether and extracted into dilute hydrochloric acid to give a dark yellow aqueous layer. The acidic layer was extracted with chloroform, basified, and extracted again with chloroform; the second chloroform layer was dried (MgSO₄) and evaporated and the colourless product (m.p. 45—50°) was crystallised from chloroform–petroleum. A purer *material* (m.p. 48—50°) was obtained by recrystallisation from chloroform–ether [Found: C, 64·1; H, 6·7; N, 18·9(7). $C_8H_{10}N_2O$ requires C, 64·0; H, 6·7; N, 18·6(5)%].

NN'-Dibenzoylbenzylhydrazine (11) is well characterised; m.p. 148,[20] 152,[21] 148—149,[22] and 149—150° (this work) (Found: C, 76·1; H, 5·4; N, 8·7. Calc. for $C_{21}H_{18}N_2O_2$: C, 76·3; H, 5·5; N, 8·5%).

Preparation of Diaroylphenylhydrazines.—Reaction of an

N-[²H₅]*Benzoyl*-N'-*benzoyl*-N-*phenylhydrazine* (15).—[²H₅]Benzoyl chloride, prepared from [²H₆]benzene *via* [²H₅]acetophenone and [²H₅]benzoic acid,[26,27] was heated under reflux with N-benzoyl-N'-phenylhydrazine (1·4 g) in chloroform (10 ml) for 5 min. The chloroform was then distilled off on a steam-bath and the residue was recrystallised from ethanol (yield 0·31 g, 34%); m.p. 178—179°. The mass spectrum (Table 2) showed that the product was about 60% [²H₅] and 30% [²H₄].

The position of the label is determined by the method of synthesis. There is good evidence that the latter acylation of phenylhydrazine does not rearrange the aroyl groups: the dimethoxy-compound (19) showed two methoxy-signals in the n.m.r. spectrum whereas the monomethoxy-compound (18) showed only one methoxy-signal. If rearrangement of aroyl groups could occur, two methoxy-peaks would be expected in the spectrum of the mono-methoxy-compound (18).

The [²H₁]-compound (14) was prepared by exchanging the

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Compounds (23) and (24) were prepared from 1,2-diphenylhydrazine (Emanuel); m.p.s 159—162° (lit.,[28,29] 161—162°) and 139—142° (lit.,[30] 136—138°), respectively. The tribenzoylhydrazine (25) melted at 157—158° (lit.,[31]

153°) (Found: C, 73·5; H, 5·1; N, 7·9. Calc. for $C_{22}H_{18}N_2O_3$: C, 73·7; H, 5·0; N, 7·8%).

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