

# MASS SPECTRA OF 2-BENZIMIDAZOLE- AND 2-BENZOXAZOLE-METHANOL, $\alpha$ , $\alpha$ -DIPHENYL

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2-Benzimidazole-methanol,  $\alpha$ ,  $\alpha$ -diphenyl and 2-benzoxazole-methanol,  $\alpha$ ,  $\alpha$ -diphenyl have been synthesised and their mass spectra studied. The study has shown some characteristic breakdown ions, and the absence or presence of these ion indicates clearly, if 1NH in a benzimidazole nucleus is substituted or not, and this information could be useful in the study of chemical constituents of natural products.

**Key words:** Mass spectra, Benzoxazole-methanol.

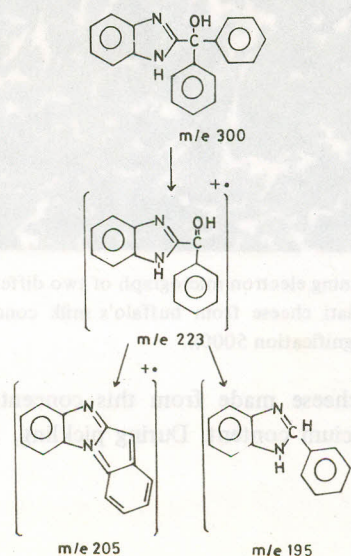
## INTRODUCTION

2-Benzimidazole-methanol,  $\alpha$ ,  $\alpha$ -diphenyl [1] (I) and 2-benzoxazole-methanol,  $\alpha$ ,  $\alpha$ -diphenyl (II) have been synthesised and their mass spectral fragmentation pattern has been studied, and some variations with some earlier observations are reported here. In case of simple tertiary alcohols, it has been found [2] that no recognisable molecular ion peak is observed, however, in case of compounds (I) and (II) significant molecular ion peaks, 59% and 51.5% of the base peak respectively, are observed. It has further been reported [3] that simpler benzimidazoles give rise to molecular ion peak, which is the base peak, however, in our present investigation it was found that compound (I) showed the expected molecular ion at  $m/e$  300, while the base peak appeared at  $m/e$  195. Initial loss of  $C_6H_5$  group followed by CO from the molecular ion gives rise to this ion (Table 1, Scheme 1). It is interesting to note that compound (II) loses CO from its molecular ion, and gives rise to a remarkable rearrangement ion at  $m/e$  273 ( $C_{19}H_{15}NO$ ;

273.1154) (Scheme II). Further loss of  $C_6H_5^+$  from it affords  $m/e$  196 ( $C_{13}H_{10}NO$ ; 196.0762) and it forms the base peak; and elimination of  $OH^+$  gives a minor peak at  $m/e$  256.  $C_6H_5^+$  is also lost from the parent ion to yield  $m/e$  224, ( $C_{14}H_{10}NO_2$ ; 224.0711, Scheme II). It is interesting to note that the normal spectrum of compound I, also shows metastable peaks centered at  $m/e$  165.7,  $m/e$  188.4 and  $m/e$  170.5 (calculated by the formula  $\frac{m_2 \times m_2}{m_1}$  when  $m_1 \rightarrow m_2$ ), which supports the fragmentation pattern shown in Scheme I. Similarly in case of compound II, metastable peaks appear in the mass spectrum at  $m/e$  247.6,  $m/e$  166.6 and  $m/e$  140.7, which further confirms the breakdown pattern as given in Scheme II. Furthermore, as has been observed previously [3] significant presence of ions at  $m/e$  205 (compound I, Table 1), could indicate the presence of a hydroxyl group at 2-position in simple benzimidazoles, when there is no substituent present at 1-position of the ring.

Table 1. Composition of some ions in the mass spectra, high resolution, of compound I.

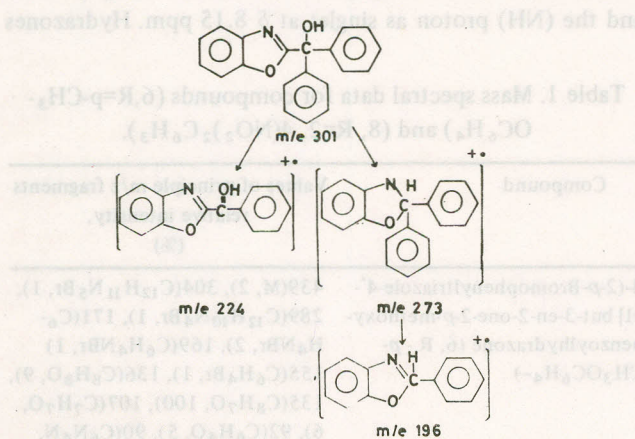
No.	$m/e$	Ion composition	Relative intensity %
1	300.1274	$C_{20}H_{16}N_2O$	59
2	223.0891	$C_{14}H_{11}N_2O$	21
3	205.0771	$C_{14}H_9N_2$	16
4	195.0936	$C_{13}H_{11}N_2$	100
5	165.0723	$C_{13}H_9$	11
6	150.0472	$C_{12}H_6$	17
7	92.0502	$C_6H_6N$	13



Scheme I. Showing breakdown of compound I.

Table 2. Composition of some ions in the mass spectra, high resolution, of compound II.

No.	m/e	Ion composition	Relative intensity %
1.	301.1112	C <sub>20</sub> H <sub>15</sub> NO <sub>2</sub>	51.5
2.	273.1154	C <sub>19</sub> H <sub>15</sub> NO	63.2
3.	256.1128	C <sub>19</sub> H <sub>14</sub> N	3.9
4.	224.0711	C <sub>14</sub> H <sub>10</sub> NO <sub>2</sub>	5.1
5.	197.0812	C <sub>13</sub> H <sub>11</sub> NO	15.1
6.	196.0762	C <sub>13</sub> H <sub>10</sub> NO	100.0
7.	195.1131	C <sub>13</sub> H <sub>9</sub> NO	16.1
8.	165.0712	C <sub>13</sub> H <sub>9</sub>	32.6
9.	105.1109	C <sub>7</sub> H <sub>5</sub> O	11.0
10.	77.0806	C <sub>6</sub> H <sub>5</sub>	15.9



Scheme II. Showing the breakdown of compound II.

## EXPERIMENTAL

Melting points were determined with a Mettler FPSI apparatus and are uncorrected. The IR spectrum was re-

corded (as mull in Nujol) with a Perkin-Elmer model 567 I.R. and U.V. spectrum was recorded with Pye-Unicam model SP8-100 spectrophotometer. N.m.r. was determined with varian model T-60 A, using tetramethylsilane as internal reference, Mass spectra were measured by the direct insertion technique with A.E.I., M.S.9 mass spectrometer.

*2-Benzimidazole, methanol,  $\alpha, \alpha$ -diphenyl.* (I) This compound was synthesised by the known [4] method.

*2-Benzoxazole methanol,  $\alpha, \alpha$ -diphenyl.* (II) Ortho-aminophenol (5:5 g) and benzoic acid (11.4 g) were refluxed in anhydrous xylene (100 ml) using a Dean and Stark apparatus. The reaction was stopped when the theoretical amount of water had been collected (ca. 5 hr). Xylene was distilled off and the resulting straw coloured solid was crystallised from ethylacetate to afford 2-benzoxazole, methanol  $\alpha, \alpha$ -diphenyl (II) as of white prisms, m.p. 107-108°; yield, 9.5 g (63 %). Mass spectrum;  $m/e$  301 ( $M$ )<sup>+</sup>; infrared spectrum indicated the characteristic band of a hydroxyl group at 3350  $cm^{-1}$ . UV  $\lambda_{Max}$ , 202, 270 nm;  $\log \epsilon$ , 4.75 and 4.34 respectively. Nmr (DMSO, D6)  $\delta$  5.22 (b, 1H, OH) removed by exchange with D<sub>2</sub>O and 6.60-7.50 (m, 14H, aromatic). (found: C, 79.75; H, 5.17; N, 4.56 % calc. for C<sub>20</sub>H<sub>15</sub>NO<sub>2</sub>: C, 79.73; H, 4.98; N, 4.65 %).

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