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# SYNTHESIS OF 11-METHYL-7,9-DIPHENYL-6H,11H-AZEPINO[2,1-*b*]-5-BENZIMIDAZOLIUM BROMIDE

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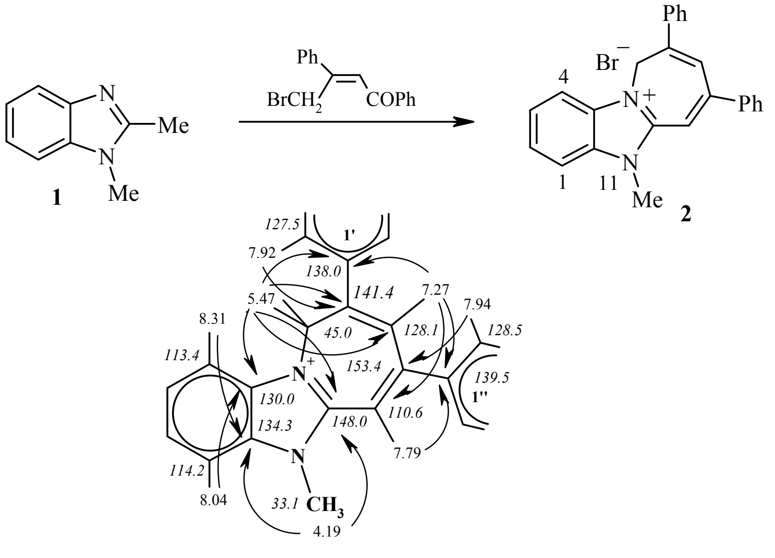
**Keywords:** azepino[2,1-*b*]benzimidazole, γ-bromodipnone, 1,2-dimethyl-1H-benzimidazole.

We have found that fusing 4-bromo-1,3-diphenyl-2-buten-1-one (γ-bromodipnone) with 1,2-dimethyl-1H-benzimidazole (**1**) leads to 11-methyl-7,9-diphenyl-6H,11H-azepino[2,1-*b*]-5-benzimidazolium bromide (**2**). We took NOESY and heteronuclear $^1H^{13}C$ HMQC and HMBC correlation spectra in addition to $^1H$ and $^{13}C$ NMR spectra to prove the structure of **2**. The experimental results are given in Table 1 and the signal assignments are shown in the scheme. The arrows show the HMBC serving as the basis of the assignments. This procedure permitted us to confirm the structure of the carbon skeleton of this molecule and determine which protons are close to each other in space. The alternation of the chemical shifts of the $^{13}C$ atoms within the seven-membered ring along the chain of atoms (from 110 to 150 ppm) suggests that the positive charge in **2** is considerably delocalized over the seven-membered ring.

There is no reliable information in the literature on the quasiaromatic azepino[2,1-*b*]benzimidazole system.



**1**

**2**

The IR spectra were taken on a Pye Unicam SP3-300 spectrometer. The $^1H$ and $^{13}C$ NMR spectra were taken on a Varian Mercury 400 spectrometer at 400 and 100 MHz, respectively, in DMSO-$d_6$ with TMS as the internal standard. The mass spectrum was taken using high-pressure liquid chromatography on an AGILENT/100-Series mass spectrometer and chemical ionization with acetonitrile and 0.05% formic acid.

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TABLE 1. $^1$H$^1$H and $^1$H$^{13}$C Correlations for Compound **2**

| $^1$H signal, δ, ppm | $^{13}$C cross peaks position | | NOESY |
|---|---|---|---|
| | HMQC | HMBC | |
| 8.31 | 113.4 | 134.3, 127.7 | 7.69, 5.47 |
| 8.04 | 114.2 | 130.6, 127.5 | 7.69, 4.19 |
| 7.94 | 128.9, 127.5 | 153.4, 141.4, 131.5, 130.6, 128.1 | 7.79, 7.57, 7.50, 7.27 |
| 7.79 | 110.6 | 153.4, 139.5, 128.1 | 7.94 |
| 7.69 | 127.5 | 130.0, 113.4, 114.2 | 8.31, 8.04 |
| 7.57 | 131.5 | 139.5 | 7.94, 7.50 |
| 7.50 | 130.0 | 138.0, 130.0 | 7.54 |
| 7.27 | 128.1 | 141.4, 139.5, 138.0, 110.6, 45.0 | 7.94 |
| 5.47 | 45.0 | 148.0, 141.4, 138.0, 130.0, 128.1 | 8.31, 7.94 |
| 4.19 | 33.1 | 148.0, 134.3 | 8.04, 7.79 |

**11-Methyl-7,9-diphenyl-6H,11H-azepino[2,1-*b*]-5-benzimidazolium Bromide (2).** A mixture of γ-bromodipnone (1.0 g, 3.32 mmol) and 1,2-dimethyl-1H-benzimidazole (0.49 g, 3.32 mmol) was fused on an oil bath at 110°C during 30 min. The melt was dissolved upon heating in 2.5 ml morpholine. The solution was cooled and 50 ml water was added. The precipitate formed was filtered off, dried, and triturated with *tert*-butyl methyl ether (10 ml). The yellow crystalline precipitate was filtered off and recrystallized from 2-propanol to give compound **2** (0.36 g, 25%); mp 275-277°C (2-propanol). IR spectrum (KBr), ν, cm$^{-1}$: 3040, 1575 (C=N), 1470, 760, 690. $^1$H NMR spectrum, δ, ppm (*J*, Hz): 8.31 (1H, m, H-4); 8.04 (1H, m, H-1); 7.94-7.90 (4H, m, H-2', H-6', H-2'', H-6''); 7.79 (1H, s, H-10); 7.69 (2H, m, H-2, H-3); 7.57 (3H, m, H-3''–H-5''); 7.51-7.45 (3H, m, H-3'–H-5'); 7.27 (1H, s, H-8); 5.47 (2H, s, H-6); 4.19 (3H, s, $CH_3$). $^{13}$C NMR spectrum, δ, ppm: 153.4 (C-9); 148.0 (C-10a); 141.4 (C-7); 139.5 (C-1''); 138.0 (C-1'); 134.3 (C-11a); 131.5 (C-4''); 130.6 (C-4'); 130.0 (C-3', C-5', C-5', C-5'', C-4a); 128.9 (C-2'', C-6''); 128.0 (C-8); 127.7 (C-2); 127.5 (C-2', C-6', C-3); 114.2 (C-1); 113.4 (C-4); 110.6 (C-10); 45.0 (C-6); 33.1 ($CH_3$). Mass spectrum, *m/z* ($I_{rel}$, %): 349.5 $[M-Br]^+$ (100). Found. %: Br 18.60; N 6.55. $C_{25}H_{21}BrN_2$. Calculated, %: Br 18.61; N 6.52.