

Phase-Transfer Catalyzed Additions; III¹. Addition of *N*-Diphenylmethylenbenzylamine to Schiff Bases

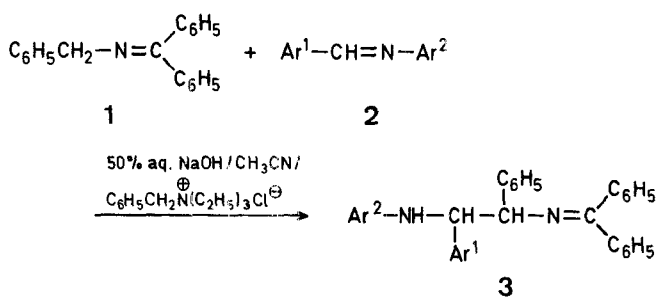
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Schiff bases, possessing a methyl or methylene group adjacent to the nitrogen atom and, therefore, generating azaallyl carbanions under basic conditions, are useful intermediates in organic synthesis²⁻⁶. However, *N*-diphenylmethylenbenzylamine **1** has been relatively little used. Its sodium salt, obtained in tetrahydrofuran in the presence of lithium dialkylamides, is known to react with butadiene giving 2,2,5-triphenyl-3-vinylpyrrolidine⁷ in 35% yield. The sodium salt, prepared in liquid ammonia by means of sodium amide⁵ or in hexamethylphosphoric triamide by sodium hydride⁶, reacts with aromatic aldehydes and aroyl halides to yield the corresponding hydroxyalkylation or aroylation derivatives, respectively.

Now we report that the carbanion of **1** can be easily generated under phase-transfer catalysis conditions using aqueous sodium hydroxide in the presence of catalytic amount of benzyltriethylammonium chloride (TEBA). The reaction of the carbanion of **1** with benzylideneaniline (**2a**) and the Schiff bases **2b-g** gives derivatives of 1,2-diaryl-1,2-ethanediamines **3a-g** (Table).

The reactions were performed at room temperature in acetonitrile solution. In the cases of **3a** and **3c**, the reaction mixtures crystallized completely in 5-10 min. In all other cases



more than 30 min were needed for complete crystallization. However, longer reaction times are not suitable because of side reactions.

Attempts to react **1** with *N*-diphenylmethylenaniline under these conditions failed.

Addition of *N*-Diphenylmethylenbenzylamine (**1**) to Schiff Bases: **2**; General Procedure:

Aqueous sodium hydroxide (50%, 3 ml) is added to the magnetically stirred solution of *N*-diphenylmethylenbenzylamine (**1**; 2.70 g, 10 mmol), the Schiff base **2** (10 mmol), and benzyltriethylammonium chloride (0.12 g, 0.5 mmol) in acetonitrile (5 ml). The mixture is stirred at room temperature for 10 min, water (100 ml) is added and the mixture is extracted with ether (3 × 30 ml). After drying with sodium sulfate, the solvent is removed and the residue is taken up in ethanol (25 ml) and reevaporated to give a solid which is recrystallized from ethanol/ethyl acetate. In the cases of **3a** and **3c**, the precipitate is filtered, washed with water until neutral, and recrystallized (Table).

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Table. 1,2-Diaryl-1,2-ethanediamines **3** prepared

Compound	Ar ¹	Ar ²	Yield [%]	m.p. [°C] ^a	Molecular formula ^b	I.R. (CHCl ₃) ν _{NH} , C=N [cm ⁻¹]	¹ H-N.M.R. (CDCl ₃ /TMS) ^c δ [ppm]
3a	C ₆ H ₅	C ₆ H ₅	71	141-143°	C ₃₃ H ₂₈ N ₂ (452.6)	3430, 1625	4.69 (d, 1H, <i>J</i> = 12 Hz); 4.73 (d, 1H, <i>J</i> = 12 Hz); 5.40 (br. s, 1H) ^d ; 6.2-7.3 (m, 25H)
3b	4-Cl-C ₆ H ₄	C ₆ H ₅	67	139-141°	C ₃₃ H ₂₇ N ₂ Cl (487.1)	3450, 1630	4.63 (d, 1H, <i>J</i> = 10.5 Hz); 4.67 (d, 1H, <i>J</i> = 10.5 Hz); 5.36 (br. s, 1H) ^d ; 6.3-7.7 (m, 24H)
3c	C ₆ H ₅	4-Cl-C ₆ H ₄	79	163-165°	C ₃₃ H ₂₇ N ₂ Cl (487.1)	3450, 1625	4.65 (d, 1H, <i>J</i> = 7.5 Hz); 4.69 (d, 1H, <i>J</i> = 7.5 Hz); 5.61 (br. s, 1H) ^d ; 6.2-7.7 (m, 24H)
3d	4-H ₃ C-C ₆ H ₄	C ₆ H ₅	20 (38) ^c	127-129°	C ₃₄ H ₃₀ N ₂ (466.6)	3450, 1630	2.28 (s, 3H); 4.66 (d, 1H, <i>J</i> = 9.75 Hz); 4.71 (d, 1H, <i>J</i> = 9.75 Hz); 5.52 (br. s, 1H) ^d ; 6.2-7.7 (m, 24H)
3e	C ₆ H ₅	4-H ₃ C-C ₆ H ₄	50 (63) ^c	160-162°	C ₃₄ H ₃₀ N ₂ (466.6)	3450, 1635	2.17 (s, 3H); 4.67 (d, 1H, <i>J</i> = 10.5 Hz); 4.71 (d, 1H, <i>J</i> = 10.5 Hz); 5.47 (br. s, 1H) ^d ; 6.2-7.7 (m, 24H)
3f	4-H ₃ CO-C ₆ H ₄	C ₆ H ₅	42	118-120°	C ₃₄ H ₃₀ N ₂ O (482.6)	3440, 1630	3.71 (s, 3H); 4.63 (d, 1H, <i>J</i> = 9.75 Hz); 4.67 (d, 1H, <i>J</i> = 9.75 Hz); 5.45 (br. s, 1H) ^d ; 6.1-7.7 (m, 24H)
3g	C ₆ H ₅	4-H ₃ CO-C ₆ H ₄	53	141-143°	C ₃₄ H ₃₀ N ₂ O (482.6)	3430, 1625	3.63 (s, 3H); 4.63 (d, 1H, <i>J</i> = 9 Hz); 4.67 (d, 1H, <i>J</i> = 9 Hz); 5.17 (br. s, 1H) ^d ; 6.1-7.7 (m, 24H)

^a Recrystallized from ethanol/ethyl acetate.

^b Satisfactory microanalyses obtained: C ± 0.19, H ± 0.11, N ± 0.18.

^c Measured with JEOL PS-100 spectrometer.

^d Disappeared after D₂O exchange.

^e Reaction time 1 h.

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