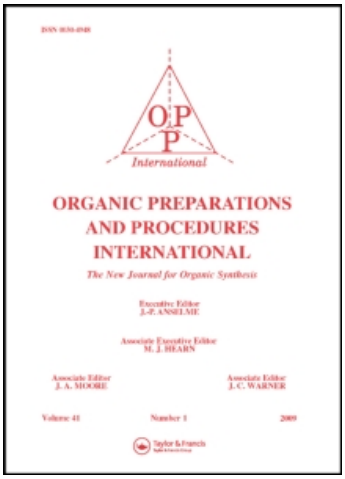

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A SIMPLE PREPARATION OF 4-AMINO-5-ARYL-2H-IMIDAZOL-2-ONES

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cm^{-1}. MS: m/z 185.

Anal. Calcd for $C_{12}H_{11}NO$: C, 77.81; H, 5.99; N, 7.56. Found: C, 77.48; H, 5.97; N, 7.48

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A SIMPLE PREPARATION OF 4-AMINO-5-ARYL-2H-IMIDAZOL-2-ONES

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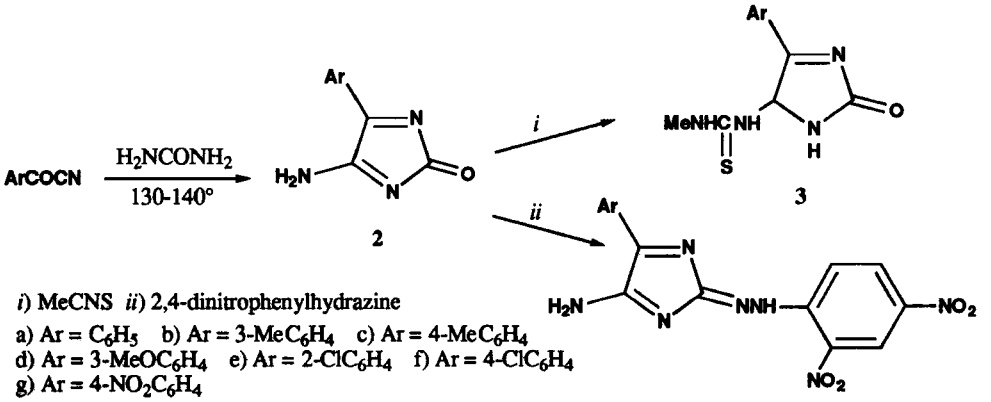
In continuation of our earlier work[1] on the utilization of α-oxonitriles **1** in heterocyclic syntheses and of our interest in imidazole chemistry, we now report a simple preparation of 4-amino-5-aryl-2H-imidazole-2-ones (**2**) by heating a mixture of **1** and urea at 130-140° for 2-6 hrs. The present synthesis affords the 4-amino-5-aryl-2H-imidazol-2-ones (**2a-g**) in 40-75% yields from **1a-g** as the only isolable products.



i) MeCNS *ii*) 2,4-dinitrophenylhydrazine
a) Ar = C_6H_5 b) Ar = 3-MeC_6H_4 c) Ar = 4-MeC_6H_4
d) Ar = 3-MeOC_6H_4 e) Ar = 2-ClC_6H_4 f) Ar = 4-ClC_6H_4
g) Ar = 4-$NO_2C_6H_4$

The structural assignment of 4-amino-5-aryl-2H-imidazol-2-one was based on spectral (UV, IR and ^{1}H NMR) and microanalytical data. The methylthioureas and 2,4-dinitrophenylhydrazones of **2a** and **2f** were prepared. The IR spectra of compounds **2a-g** (nujol) show medium intensity absorptions at 3350-3200 (NH_2), 1720-1700 (CO) and 1620-1600 cm^{-1}. The UV of **2a**, **2b** and **2d** in ethanol exhibits peaks in the range 276-279 nm (log ε 3.28-3.10), 233-236 nm (log ε 4.38-4.06) and 209-210 nm (log ε 4.14-4.02).

TABLE 1. Yields and Physical Constants of 2-Imidazolones **2**[a]

Compd	mp. (°C)	Yield (%)	Time (hrs)	^{1}H NMR (δ)	Elemental Analyses (Found) C	H	N
2a	206	75	2	7.23-8.19 (m, 5H, Ar-H) 10.53 (s, 2H, NH)	62.40 (62.10)	4.05 (4.17)	24.27 (24.41)
2b	165-166	70	3	2.33 (s, 3H, CH_3), 7.14-8.16 (m, 4H, Ar-H), 10.38 (s, 2H, NH) D_2O-exchangeable	64.17 (63.90)	4.81 (5.10)	22.46 (22.54)
2c	190	70	4		64.17 (64.25)	4.81 (5.10)	22.46 (22.76)
2d	212	65	6	3.25 (s, 3H, OCH_3), 7.16-8.00 (m, 4H, Ar-H) 10.23(broad, 2H, NH) D_2O-exchangeable	59.11 (58.90)	4.43 (4.67)	20.68 (20.38)
2e	160	40	6	6.66-7.93 (m, 4N, Ar-H) 8.60 (s, 2H, NH), D_2O-exchangeable	52.04 (51.81)	2.89 (3.02)	20.24 (20.54)
2f	242	70	3	6.85-8.05 (m, 4H, Ar-H), 1.57 (s, 2H, NH) ppm	52.04 (52.15)	2.89 (2.92)	20.14 (20.24)
2g	120	70	3	8.14-9.14 (m, 4H, Ar-H), 12.16 (s, 2H, NH) ppm	49.54 (49.40)	2.77 (2.65)	25.68 (25.40)

a) Except for colorless **2e**, all compounds are yellow and were crystallized from ethanol.

EXPERIMENTAL SECTION

Mps were obtained on a Gallenkamp apparatus and are uncorrected. The purity of compounds was routinely checked by TLC using silica gel G (E. Merck). Elemental analyses (C, H and N) were performed by a Coleman Analyser. The IR spectra were recorded with a Perkin Elmer 783 grating spectrophotometer as nujol mulls. The ^{1}H NMR spectra were run on JEOL FT-NMR Fx-90Q spectrometer at the probe temperature (27°) in dimethyl sulfoxide-d_6 solution using TMS as an internal standard. UV spectra were recorded on Cary-2390 and Shimadzu UV-160 A model UV-visible spectrometers. The α-oxonitriles **1a-g** were prepared by reported methods.[3]

4-Amino-5-phenyl-2H-imidazole-2-one (2a). Typical Procedure.- A mixture of (1.20 g, 0.02 mole)

of urea and benzoyl cyanide (2.62 g, 0.02 mole) was heated on an oil-bath at 130-140° for 2 hrs. The solid obtained on cooling was recrystallized from ethanol to give 2.60 g (70%) of light yellow needles, mp. 204-206°.

1-Methyl-3-(5-phenyl-2*H*-imidazol-2-one-4-yl-2-thiourea) (3a). Typical Procedure.- 4-Amino-5-phenyl-2H-imidazol-2-one (**2a**, 1.73 g, 0.01 mole) was heated with methyl isothiocyanate (0.75 g, 0.01 mole) in dry benzene (20 mL) for 1 hr on a steam bath, a solid mass was formed. The solvent was evaporated and the residue washed with 50% aqueous ethanol.

Compound 3a, colorless solid, mp. 221° (EtOH)

Anal. Calcd for $C_{11}H_{10}N_4OS$: C, 53.65; H, 4.06; N, 22.76. Found: C, 53.95; H, 3.76; N, 22.86

Compound 3f, colorless solid, mp. 290° (EtOH)

Anal. Calcd for $C_{11}H_9N_4ClOS$: C, 47.55; H, 3.28; N, 19.96. Found: C, 47.35; H, 3.48; N, 19.80

2,4-Dinitrophenylhydrazones (4a). Typical Procedure.- A solution of 0.86 g (0.005 mole) of 4-amino-5-phenyl-2*H*-imidazol-2one in 30 mL ethanol was treated with freshly prepared 2,4-dinitrophenylhydrazine solution (ethanol) at 25° for 2 hrs; the yellow precipitate was collected, crystallized from ethanol, 0.90 g (50%).

Compound 4a, orange crystals, mp. 180°

Anal. Calcd for $C_{15}H_{11}N_7O_4$: C, 50.99; H, 3.15; N, 27.76. Found: C, 50.69; H, 3.35; N, 27.60

Compound 4f, yellow crystals, mp. 210° (EtOH)

Anal. Calcd for $C_{15}H_{10}ClN_7O_4$: C, 46.45; H, 2.55; N, 25.29. Found: C, 46.25; H, 2.50; N, 25.50

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