Chloramine-T mediated synthesis of 6-aryl-9-(pyridine-4-yl)[1,2,4]triazolo[4,3-*a*][1,8]naphthyridines

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Abstract- The reaction between The reaction between 3-aryl-2-hydrazino-1,8-naphthyridines1 with 4-pyridinecarboxaldehyde **2** in the presence of catalytic amount of DMF under microwave irradiation afforded the respective 3-phenyl-2-(2-(pyridin-4-ylmethylene)hydrazinyl)-1,8-naphthyridine **3** in excellent yields. Oxidative cyclization of hydrazones **3** with chloramine-T in methanol under microwave irradiation resulted in the formation of 6 Aryl-9-(pyridin-4-yl)-[1,2,4]triazolo[4,3-*a*][1,8]naphthyridines **4**. The oxidative transformation is clean and efficient. The experimental procedure is very simple. with excellent yields. (**Scheme I**) and Table-1,2. The structures of the compounds were elucidated by spectral analysis.

Keywords- 1,8-naphthyridines, chloramine-T, MEOH, DMF, MWI.

1. INTRODUCTION

1,8-Naphthyridine and its derivatives as an important class of nitrogen containing heterocyclic compounds play a significant role in the organic synthetic chemistry due to their various biological activities.¹⁻³ Various 1,2,4-triazoles have been extensively explored for their applications in the field of activities.4-6 biological and pharmacological Therefore, 1,8-naphthyridine and 1,2,4-triazole have broad range of antimicrobial spectrum and have privileged nuclei to display medicinal activities. Microwave-assisted organic synthesis has attracted considerable attention in recent years⁷⁻¹⁰, due to enhanced reaction rates, high yields, improved selectivity and cleaner products. Several methods have been developed for performing reactions with microwave irradiation in solution and under solventfree conditions, but a homogeneous mixture is preferred to obtain uniform heating. The solvents with higher dielectric constants are superheated and the reactions take place rapidly. Chloramine-T (CAT) is a very versatile oxidizing agent and is of much importance in its synthetic utility.^{11,12}

In continuation to our earlier work and varied biological activities exhibited by 1,8-

naphthyridines, we here by reports. the synthesis of 1,2,4-triazolo[4,3-a] [1,8] naphthyridines using chloramine-T in methanol under microwave irradiation. The synthetic route to these compounds is profiled in **Scheme I**. The starting compounds, 3-aryl-2-hydrazino-1,8-naphthyridines **1** were prepared according to our reported procedures¹³⁻²⁰.

2. EXPERIMENTAL SECTION

Melting points were determined on a Cintex melting point apparatus and are uncorrected. The ¹H NMR spectra were recorded on a BRUKER Spectrometer (400 MHz). Chemical shifts were reported in parts per million using tetramethylsilane as an internal standard and were given in δ units. The solvent for NMR spectra was DMSO.

Infrared spectra were taken on SHIMADZU-FTIR-8400 Spectrophotometer instrument in the frequency range of 4000-400 cm-1 by KBr powder method. The Mass spectra were recorded by MS-SHIMADZU-QP2010. All reactions were monitored by thin layer chromatography, carried out on 0.2 mm silica gel 60F254 (Merck) plates using UV light for detection. Common reagent grade chemicals are either commercially available and were used without further purification.



k 4-CH₃C₆H₄

1 Naphthyl

Scheme I

 $4-ClC_6H_4$

 $2-FC_6H_4$

e,

f,

3. RESULTS AND DISCUSSION

Condensation of 3-aryl-2-hydrazino-1,8naphthyridines **1** with 4-pyridinecarboxaldehyde **2** in the presence of catalytic amount of DMF under microwave irradiation afforded the respective 3phenyl-2-(2-(pyridin-4-ylmethylene)hydrazinyl)-1,8naphthyridine **3** in excellent yields.

Oxidative cyclization of hydrazones **3** with chloramine-T in methanol under microwave irradiation resulted in the formation of desired product with an excellent yields namely 6 Aryl-9-(pyridin-4-yl)-[1,2,4]triazolo[4,3

a][1,8]naphthyridines **4**.The oxidative transformation is clean and efficient.

The experimental procedure is very simple. The high yield transformation did not form any undesirable by-products. Furthermore, the products were obtained with a higher degree of purity by this procedure and in most cases no further purification was needed.

Interestingly, this oxidative reaction proceeds only to a minor extent (5-8% in 3.5-4.5 min) when conducted under conventional conditions in an oil-bath preheated to 110° C (temperature measured at the end of exposure during microwave experiment) which confirms the rate augmentation during microwave heating.

The structural assignments of compounds **3** and **4** were based on their spectroscopic (IR, ¹H NMR and mass) and analytical data.

General procedure for the synthesis of 3-Aryl-2-(2-(pyridin-4-ylmethylene)hydrazinyl)-1,8naphthyridine 3

A mixture of **1** (0.01 mole) Pyridinecarboxaldehyde **2** (0.01 mole) and DMF (5 drops) was subjected to microwave irradiation at 400 W intermittently at 30 sec intervals for the specified time (**Table I**). On completion of reaction, as monitored by TLC, the reaction mixture was cooled and treated with cold water. The resulting solid product was filtered, washed with water and recrystallized from ethanol to give **3**.

3a: IR (KBr): 3353 (NH), 1623 (C=N); ¹H NMR (CDCl₃): 8.78 (m, 1H, C₇-H of naphthyridine), 8.60 (d, 2H, C₂-H, C₆-H of pyridine), 8.26 (d, 2H C₃-H, C₅-H of pyridine), 8.08 (s, 1H, C₄-H of naphthyridine), 7.28-7.75 (m, 7H, C₅-H, C₆-H of naphthyridine, 5Ar-H), 8.83 (s, 1H, N=CH), 10.32(s, 1H, NH); MS(ES⁺): m/z 326.25 [M+H]⁺.

3b: IR (KBr): 3342 (NH), 1614 (C=N); ¹H NMR (CDCl₃): 3.86 (s, 3H, OCH₃), 8.76 (m, 1H, C₇-H of naphthyridine), 8.58 (d, 2H, C₂-H, C₆-H of pyridine), 8.25 (d, 2H C₃-H, C₅-H of pyridine), 8.04 (s, 1H, C₄-H of naphthyridine), 7.25-7.70 (m, 6H, C₅-H, C₆-H of naphthyridine, 4Ar-H), 8.79 (s, 1H, N=CH), 10.29(s, 1H, NH); MS(ES⁺): m/z 356.30 [M+H]⁺.

3c: IR (KBr): 3360 (NH), 1624 (C=N); ¹H NMR (CDCl₃): 8.82 (m, 1H, C₇-H of naphthyridine), 8.63 (d, 2H, C₂-H, C₆-H of pyridine), 8.29 (d, 2H C₃-H, C₅-H of pyridine), 8.12 (s, 1H, C₄-H of naphthyridine), 7.31-7.78 (m, 7H, C₅-H, C₆-H of naphthyridine, 4Ar-H), 8.85 (s, 1H, N=CH), 10.35(s, 1H, NH); MS(ES⁺): m/z 360.10 [M+H]⁺.

3d: IR (KBr): 3357 (NH), 1625 (C=N); ¹H NMR (CDCl₃): 8.78 (m, 1H, C₇-H of naphthyridine), 8.60 (d, 2H, C₂-H, C₆-H of pyridine), 8.26 (d, 2H C₃-H, C₅-H of pyridine), 8.08 (s, 1H, C₄-H of naphthyridine), 7.28-7.75 (m, 7H, C₅-H, C₆-H of naphthyridine, 5Ar-H), 8.83 (s, 1H, N=CH), 10.32(s, 1H, NH); MS(ES⁺): m/z 360.13 [M+H]⁺.

3e: IR (KBr): 3351 (NH), 1627 (C=N); ¹H NMR (CDCl₃): 8.81 (m, 1H, C₇-H of naphthyridine), 8.62 (d, 2H, C₂-H, C₆-H of pyridine), 8.28 (d, 2H C₃-H, C₅-H of pyridine), 8.11 (s, 1H, C₄-H of naphthyridine), 7.30-7.79 (m, 7H, C₅-H, C₆-H of naphthyridine, 4Ar-H), 8.84 (s, 1H, N=CH), 10.33(s, 1H, NH); MS(ES⁺): m/z 360.13 [M+H]⁺.

3f: IR (KBr): 3362 (NH), 1624 (C=N); ¹H NMR (CDCl₃): 8.82 (m, 1H, C₇-H of naphthyridine), 8.65 (d, 2H, C₂-H, C₆-H of pyridine), 8.28 (d, 2H C₃-H, C₅-H of pyridine), 8.15 (s, 1H, C₄-H of naphthyridine), 7.34-7.83 (m, 7H, C₅-H, C₆-H of naphthyridine, 4Ar-H), 8.85 (s, 1H, N=CH), 10.31(s, 1H, NH); MS(ES⁺): m/z 344.13 [M+H]⁺.

3g: IR (KBr): 3358 (NH), 1623 (C=N); ¹H NMR (CDCl₃): 8.81 (m, 1H, C₇-H of naphthyridine), 8.64 (d, 2H, C₂-H, C₆-H of pyridine), 8.29 (d, 2H C₃-H, C₅-H of pyridine), 8.15 (s, 1H, C₄-H of naphthyridine), 7.33-7.84 (m, 7H, C₅-H, C₆-H of naphthyridine, 4Ar-H), 8.84 (s, 1H, N=CH), 10.32(s, 1H, NH); MS(ES⁺): m/z 344.25 [M+H]⁺.

3h: IR (KBr): 3350 (NH), 1625 (C=N); ¹H NMR (CDCl₃): 8.79 (m, 1H, C₇-H of naphthyridine), 8.63 (d, 2H, C₂-H, C₆-H of pyridine), 8.27 (d, 2H C₃-H, C₅-H of pyridine), 8.09 (s, 1H, C₄-H of

naphthyridine), 7.32-7.89 (m, 7H, C₅-H, C₆-H of naphthyridine, 5Ar-H), 8.83 (s, 1H, N=CH), 10.31(s, 1H, NH); $MS(ES^+)$: m/z 344.25 [M+H]⁺.

3i: IR (KBr): 3363 (NH), 1626 (C=N); ¹H NMR (CDCl₃): 8.86 (m, 1H, C₇-H of naphthyridine), 8.61 (d, 2H, C₂-H, C₆-H of pyridine), 8.28 (d, 2H C₃-H, C₅-H of pyridine), 8.09 (s, 1H, C₄-H of naphthyridine), 7.38-7.86 (m, 7H, C₅-H, C₆-H of naphthyridine, 5Ar-H), 8.86 (s, 1H, N=CH), 10.32(s, 1H, NH); MS(ES⁺): m/z 394.23 [M+H]⁺.

3j: IR (KBr): 3365 (NH), 1628 (C=N); ¹H NMR (CDCl₃8.75 (m, 1H, C₇-H of naphthyridine), 8.63 (d, 2H, C₂-H, C₆-H of pyridine), 8.29 (d, 2H C₃-H, C₅-H of pyridine), 8.11 (s, 1H, C₄-H of naphthyridine), 7.38-7.96 (m, 7H, C₅-H, C₆-H of naphthyridine, 5Ar-H), 8.86 (s, 1H, N=CH), 10.35(s, 1H, NH); MS(ES⁺): m/z 394.23 [M+H]⁺.

3k: IR (KBr): 3345 (NH), 1612 (C=N); ¹H NMR (CDCl₃): 2.42(s, 3H, CH₃), 8.76 (m, 1H, C₇-H of naphthyridine), 8.61 (d, 2H, C₂-H, C₆-H of pyridine), 8.23 (d, 2H C₃-H, C₅-H of pyridine), 8.09 (s, 1H, C₄-H of naphthyridine), 7.23-7.70 (m, 7H, C₅-H, C₆-H of naphthyridine, 4Ar-H), 8.83 (s, 1H, N=CH), 10.32(s, 1H, NH); MS(ES⁺): m/z 340.27 [M+H]⁺.

3I: IR (KBr): 3348 (NH), 1616 (C=N); ¹H NMR (CDCl₃): 8.79 (m, 1H, C₇-H of naphthyridine), 8.60 (d, 2H, C₂-H, C₆-H of pyridine), 8.26 (d, 2H C₃-H, C₅-H of pyridine), 8.09 (s, 1H, C₄-H of naphthyridine), 7.28-7.83 (m, 11H, C₅-H, C₆-H of naphthyridine, 9Ar-H), 8.84 (s, 1H, N=CH), 10.30(s, 1H, NH); MS(ES⁺): m/z 376.27 [M+H]⁺.

General procedure for the synthesis of 6-Aryl-9-(pyridin-4-yl)-[1,2,4]triazolo[4,3a][1,8]naphthyridine 4

To a solution of appropriate hydrazone **3** (0.01 mole) in methanol (15 ml), chloramine-T (20.0 mmole) was added. The reaction mixture was exposed to microwaves at 400 W intermittently at 30 sec intervals for specified time (**Table II**). After complete conversion as indicated by TLC, the reaction mixture was cooled to RT and digested with cold water. The solid thus obtained was filtered, washed with water and recrystallized from ethanol to afford **4**.

4a: IR (KBr): 1612 (C=N); ¹H NMR (CDCl₃): 8.75 (m, 1H, C₂-H of naphthyridine) 8.56 (s, 1H, C₅-H of naphthyridine), 8.30 (d, 2H, C₂-H, C₆-H of pyridine),

8.23(d, C₃-H, C₅-H of pyridine), 7.98 (m, 1H, C₄-H of naphthyridine), 7.52-7.82 (m, 6H, C₃-H of naphthyridine, 5Ar-H); $MS(ES^+)$: *m/z* 324.25[M+H]⁺.

4b: IR (KBr): 1598 (C=N); ¹H NMR (CDCl₃): 3.95(s,3H,OCH₃) 8.73(m, 1H, C₂-H of naphthyridine) 8.54(s,1H, C₅-H of naphthyridine), 8.27 (d, 2H, C₂-H, C₆-H of pyridine), 8.21(d, C₃-H, C₅-H of pyridine), 7.97 (m, 1H, C₄-H of naphthyridine), 7.49-7.79 (m, 5H, C₃-H of naphthyridine, 4Ar-H); MS(ES⁺): m/z 354.30[M+H]⁺.

4c: IR (KBr): 1614 (C=N); ¹H NMR (CDCl₃): 8.78 (m, 1H, C₂-H of naphthyridine)8.57 (s, 1H, C₅-H of naphthyridine), 8.42 (d, 2H, C₂-H, C₆-H of pyridine), 8.24(d, C₃-H, C₅-H of pyridine), 8.11 (m, 1H, C₄-H of naphthyridine), 7.59-7.88 (m, 5H, C₃-H of naphthyridine, 4Ar-H);MS(ES⁺): m/z 358.31[M+H]⁺.

4d: IR (KBr): 1611 (C=N); ¹H NMR (CDCl₃): 8.77 (m, 1H, C₂-H of naphthyridine)8.57 (s, 1H, C₅-H of naphthyridine), 8.32 (d, 2H, C₂-H, C₆-H of pyridine), 8.25(d, C₃-H, C₅-H of pyridine), 8.00 (m, 1H, C₄-H of naphthyridine), 7.58-7.85 (m, 5H, C₃-H of naphthyridine, 4Ar-H); MS(ES⁺): m/z 358.31[M+H]⁺.

4e: IR (KBr): 1624 (C=N); ¹H NMR (CDCl₃): 8.76 (m, 1H, C₂-H of naphthyridine)8.58 (s, 1H, C₅-H of naphthyridine), 8.31 (d, 2H, C₂-H, C₆-H of pyridine), 8.25(d, C₃-H, C₅-H of pyridine), 7.99 (m, 1H, C₄-H of naphthyridine), 7.56-7.83 (m, 5H, C₃-H of naphthyridine, 4Ar-H); MS(ES⁺): m/z 358.31[M+H]⁺.

4f: IR (KBr): 1613 (C=N); ¹H NMR (CDCl₃): 8.81 (m, 1H, C₂-H of naphthyridine)8.63 (s, 1H, C₅-H of naphthyridine), 8.35 (d, 2H, C₂-H, C₆-H of pyridine), 8.27(d, C₃-H, C₅-H of pyridine), 8.15 (m, 1H, C₄-H of naphthyridine), 7.57-7.91 (m, 5H, C₃-H of naphthyridine, 4Ar-H);: m/z 342.19[M+H]⁺.

4g: IR (KBr): 1610 (C=N); ¹H NMR (CDCl₃): 8.79 (m, 1H, C₂-H of naphthyridine)8.58 (s, 1H, C₅-H of naphthyridine), 8.35 (d, 2H, C₂-H, C₆-H of pyridine), 8.28(d, C₃-H, C₅-H of pyridine), 8.13 (m, 1H, C₄-H of naphthyridine), 7.62-7.98 (m, 5H, C₃-H of naphthyridine,4Ar-H); MS(ES⁺): m/z 342.19[M+H]⁺.

4h: IR (KBr): 1625 (C=N); ¹H NMR (CDCl₃): 8.81 (m, 1H, C_2 -H of naphthyridine)8.63 (s, 1H, C_5 -H of naphthyridine), 8.31 (d, 2H, C_2 -H, C_6 -H of pyridine),

8.24(d, C₃-H, C₅-H of pyridine), 8.13 (m, 1H, C₄-H of naphthyridine), 7.61-7.95 (m, 5H, C₃-H of naphthyridine, 4Ar-H); $MS(ES^+)$: m/z 342.19[M+H]⁺.

4i: IR (KBr): 1609 (C=N); ¹H NMR (CDCl₃): 8.81 (m, 1H, C₂-H of naphthyridine)8.65 (s, 1H, C₅-H of naphthyridine), 8.39 (d, 2H, C₂-H, C₆-H of pyridine), 8.28(d, C₃-H, C₅-H of pyridine), 8.13 (m, 1H, C₄-H of naphthyridine), 7.68-7.99 (m, 5H, C₃-H of naphthyridin , 4Ar-H); MS(ES⁺): m/z 392.23[M+H]⁺.

4j: IR (KBr): 1612 (C=N); ¹H NMR (CDCl₃): 8.83 (m, 1H, C₂-H of naphthyridine)8.63 (s, 1H, C₅-H of naphthyridine), 8.40 (d, 2H, C₂-H, C₆-H of pyridine), 8.30(d, C₃-H, C₅-H of pyridine), 8.11 (m, 1H, C₄-H of naphthyridine), 7.66-7.95 (m, 5H, C₃-H of naphthyridin ,4Ar-H); MS(ES⁺): m/z 392.13[M+H]⁺.

4k: IR (KBr): 1605 (C=N); ¹H NMR (CDCl₃): 2.54(s,3H,CH₃) 8.73 (m, 1H, C₂-H of naphthyridine)8.55 (s, 1H, C₅-H of naphthyridine), 8.29 (d, 2H, C₂-H, C₆-H of pyridine), 8.21(d, C₃-H, C₅-H of pyridine), 7.95 (m, 1H, C₄-H of naphthyridine), 7.51-7.79 (m, 5H, C₃-H of naphthyridin, 4Ar-H); MS(ES⁺): m/z 338.27[M+H]⁺.

41: IR (KBr): 1615 (C=N); ¹H NMR (CDCl₃): 8.76 (m, 1H, C₂-H of naphthyridine)8.58 (s, 1H, C₅-H of naphthyridine), 8.32 (d, 2H, C₂-H, C₆-H of pyridine), 8.26(d, C₃-H, C₅-H of pyridine), 7.99 (m, 1H, C₄-H of naphthyridine), 7.52-8.08 (m, 10H, C₃-H of naphthyridin, 9Ar-H); MS(ES⁺): m/z 374.19[M+H]⁺.

	•	•	•				•	
Compd	Ar	Reaction time	m.p. °C	Yield (%)	Mol. Formula	Found (%) (Calcd)		
		(min)				С	Н	Ν
39	CeHe	15	192	92	CarHusNs	73.96	4 66	21.57
Ju	0,115	1.5	172	2	020115115	(73.83	4.65	21.52)
3b	$3-CH_3OC_6H_4$	2.0	178	94	$C_{21}H_{17}N_5O$	71.11	4.83	19.76
						(70.97	4.82	19.71)
3c	$2-ClC_6H_4$	2.0	145	93	$C_{20}H_{14}ClN_5$	66.90	3.94	19.51
						(66.76	3.92	19.46)
3d	$3-ClC_6H_4$	1.5	172	92	$C_{20}H_{14}CIN_5$	66.88	3.93	19.50
	0 +				20 14 3	(66.76	3.92	19.46)
3 e	$4-ClC_6H_4$	2.5	205	96	$C_{20}H_{14}ClN_5$	66.89	3.94	19.52
						(66.76	3.92	19.46)
3f	2-FC ₄ H ₄	2.0	280	93	C20H14FN5	70 10	4 13	20.45
~		2.0	200	20	-201415	(69.96	4.11	20.40)
3g	$3-FC_6H_4$	2.0	256	94	$C_{20}H_{14}FN_5$	70.08	4.12	20.46
						(69.96	4.11	20.40)

Table I — Physical and analytical data of 3-Aryl-2-(2-(pyridin- 4-ylmethylen

in- 4-ylmethylene)hydrazinyl)-1,8-naphthyridine **3**

3h	$4-FC_6H_4$	1.5	225	94	$C_{20}H_{14}FN_5$	70.09 (69.96	4.12 4.11	20.44 20.40)
3i	$2\text{-}CF_3C_6H_4$	2.0	162	95	$C_{21}H_{14}F_{3}N_{5} \\$	64.26 (64.12	3.60 3.59	17.85 17.80)
3ј	$4-CF_3C_6H_4$	2.5	163	93	$C_{21}H_{14}F_{3}N_{5} \\$	64.26 (64.11	3.60 3.59	17.85 17.80)
3k	4- $CH_3C_6H_4$	1.5	178	94	$C_{21}H_{17}N_5$	71.11 (70.97	4.83 4.82	25.76 25.71)
31	Naphthyl	2.0	189	93	$C_{24}H_{17}N_5$	73.96 (73.83	4.66 4.65	21.57 21.52)

Table II — Physical and analytical data of 6 Aryl-9-(pyridin-4-yl)- [1,2,4]triazolo[4,3-a][1,8]naphthyridine 4

Compd	Ar	Reaction time	m.p. °C	Yield (%)	Mol. Formula	Found (%) (Calcd)		Calcd)
		(min)				С	Н	Ν
4 a	C ₆ H ₅	6.0	235	85	$C_{20}H_{13}N_5$	74.41	4.07	21.70
						(74.29	4.05	21.66)
4b	3-	5.0	220	87	$C_{21}H_{15}N_5O$	71.51	4.30	19.86
	$CH_3OC_6H_4$					(71.38	4.28	19.82)
4 c	$2-ClC_6H_4$	5.5	195	86	$C_{20}H_{12}ClN_5$	67.25	3.40	19.63
						(67.14	3.38	19.57)
4d	$3-ClC_6H_4$	6.0	223	85	$C_{20}H_{12}ClN_5$	67.27	3.40	19.62
						(67.14	3.38	19.57)
4 e	$4-ClC_6H_4$	6.0	210	88	$C_{20}H_{12}ClN_5$	67.26	3.39	19.61
						(67.14	3.38	19.57)
4f	$2-FC_6H_4$	5.5	223	87	$C_{20}H_{12}FN_5$	70.50	3.56	20.57
						(70.37	3.54	20.52)
4g	$3-FC_6H_4$	6.0	245	86	$C_{20}H_{12}FN_5$	70.51	3.55	20.56
						(70.37	3.54	20.52)
4h	$4 - FC_6H_4$	6.0	248	87	$C_{20}H_{12}FN_5$	70.42	3.56	20.58
						(70.37	3.54	20.52)
4i	$2-CF_3C_6H_4$	5.5	246	89	$C_{21}H_{12}F_3N_5$	64.58	3.11	17.94
						(64.45	3.09	17.90)
4j	$4-CF_3C_6H_4$	5.5	246	86	$C_{21}H_{12}F_3N_5$	64.58	3.11	17.94
						(64.45	3.09	17.90)
4 k	$4-CH_3C_6H_4$	6.0	220	87	$C_{21}H_{15}N_5$	71.51	4.30	24.86
						(71.38	4.28	24.82)
41	Naphthyl	6.5	235	86	$C_{24}H_{15}N_5$	74.41	4.07	21.70
						(74.29	4.05	21.66)

4. CONCLUSION

The significant advantages of this procedure are operational simplicity, short reaction time, pure products, inexpensive and non-toxicity of the reagent and high yields.

ACKNOWLEDGEMENT

The authors are thankful to the Director, IICT, Hyderabad for providing ¹H NMR and mass spectral data. One of them(P K Ch) is grateful to UGC, New Delhi for the award of a Senior Research Fellowship.

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