

A Green, Chemoselective, and Efficient Protocol for Paal–Knorr Pyrrole and Bispyrrole Synthesis Using Biodegradable Polymeric Catalyst PEG-SO₃H in Water

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ABSTRACT: In water and in the presence of 1 mol % of Poly(ethylene glycol)-bounded sulfonic acid, amines undergo smooth condensation with 2,5-hexadione at room temperature to afford the corresponding pyrroles in good to excellent yields. The sole products were solidified as the reaction proceeded and separated by simple filtration. In addition, bispyrroles were synthesized in excellent

yields via this eco-friendly protocol. The method is easy, simple, cost-effective, chemoselective, and environmentally benign that introduces a beautiful example of click chemistry in water. © 2012 Wiley Periodicals, Inc. *J Appl Polym Sci* 125: 1339–1345, 2012

Key words: water; Paal–Knorr; PEG-SO₃H; pyrrole; amine

INTRODUCTION

Carrying out organic reactions in water has become highly desirable in recent years to meet environmental considerations.^{1–14} The replacement of expensive, toxic, flammable, and not recyclable organic solvents with water as a cheap, abundant, nontoxic, nonflammable, and green solvent is desirable, especially for “Click Chemistry.”¹ In addition, water with its chemical and physical properties imposes selectivity and reactivity in reactions conducted in aqueous media, which cannot be gained using organic solvents.^{6–9} However, dissolving many organic compounds in water is difficult and for this reason, reactions cannot proceed easily in aqueous media.¹⁰ One way to improve the solubility of substrates in water is the use of surface-active reagents that can form micelles or vesicular cavities.^{11–14} Poly(ethylene glycol)-bounded sulfonic acid (PEG-SO₃H) is a soluble polymeric catalyst to fulfill this aim by both dissolving the organic compounds in water and catalyzing the reaction by Brønsted acid sites.^{15–17}

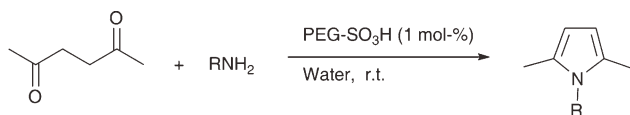
The pyrrole ring system is a useful structural element in medicinal chemistry¹⁸ and has found broad application in drug development as an antibacterial, antiviral, anti-inflammatory, antitumoral, and anti-

oxidant agent.¹⁹ Pyrrole derivatives are a highly versatile class of intermediates in the synthesis of natural products, heterocyclic compounds²⁰ and are widely used in materials science.²¹ It is, therefore, not surprising that many synthetic methods have been developed for the preparation of these compounds.²² Despite these developments, the Paal–Knorr²¹ reaction is considered to be the most attractive method for the synthesis of pyrroles; wherein 1,4-dicarbonyl compounds are converted to pyrroles by reacting with primary amines in the presence of various promoting agents.^{23–40} However, some of these methods suffer from certain drawbacks such as using hazardous organic solvents, high cost, using a stoichiometric or relatively expensive catalyst, tedious workup leading to the generation of large amounts of toxic metal-containing wastes and low yields of the products. Therefore, the development of facile and environmentally benign methods for the synthesis of pyrroles is of interest. The search continues for finding a better catalyst in terms of operational simplicity, economic viability, and greater selectivity.

Using water as a reaction medium has been one of our interests in recent years. Along this line, we have successfully used water and micellar solutions for Mannich, Biginelli, Hantzsch, Michael, and some other synthetic transformations.^{41–44} Now, we wish to report PEG-SO₃H as a soluble polymeric catalyst that readily catalyzes Paal–Knorr pyrrole and bispyrrole synthesis in water (Scheme 1). The solid PEG-SO₃H,^{15–17} easily prepared from available cheap starting materials, is not a corrosive compound, its handling is easy and does not need special precautions.

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Scheme 1 PEG-SO₃H catalyzed Paal-Knorr pyrrole synthesis in water.

EXPERIMENTAL

General remarks

Chemicals were purchased from Merck and Fluka Chemical Companies. All the products are known and were characterized by comparing of their physical data with those reported in the literature. IR spectra were run on a Shimadzu model 8300 Fourier transform infrared (FTIR) spectrophotometer. Nuclear magnetic resonance (NMR) spectra were recorded on a Bruker Avance DPX-400 and 500. The purity of the products and the progress of the reactions were achieved and established by thin layer chromatography (TLC) on silica-gel polygram SILG/UV₂₅₄ plates or by a Shimadzu Gas Chromatograph GC-14A instrument with a flame ionization detector.

General procedure for the condensation of amine to 2,5-hexanedione

PEG-SO₃H (0.12 g, 0.02 mmol) was added to a mixture of amine (2.0 mmol) and 2,5-hexanedione (0.28

g, 2.4 mmol). The reaction mixture was stirred at room temperature for the appropriate time (Tables I and II). During the stirring, a desired solid product separated out. After the completion of the reaction (monitored by TLC), water (10 mL) was added to the reaction mixture. The solid product was collected by a simple filtration, washed with water (4 mL), and air-dried to provide the sole product as a white or cream powder.

The selected spectral and analytical data for new compounds

1-(3-(2,5-Dimethyl-1*H*-pyrrol-1-yl)phenyl)ethanone (Table II, entry 10): white solid; m.p.: 64–66°C; FTIR: ν_{\max} = 2930, 1681, 1588, 1485, 1437, 1256, 1216, 797, 760, 692 cm⁻¹; ¹H-NMR (400 MHz, CDCl₃): δ = 2.05 (s, 6H), 2.64 (s, 3H), 5.94 (s, 2H), 7.45 (d, *J* = 8 Hz, 1H), 7.59 (t, *J* = 8 Hz, 1H), 7.82 (s, 1H), 8.01 (d, *J* = 7.6 Hz, 1H) ppm; ¹³C-NMR (100 MHz, CDCl₃): δ = 13.1, 26.8, 106.3, 127.5, 128.1, 128.7, 129.5, 132.1, 138.2, 139.4, 197.1 ppm.

1-(4-Fluorobenzyl)2,5-dimethyl-1*H*-pyrrole (Table II, entry 13): white solid; m.p.: 54–56°C; FTIR: ν_{\max} = 3010, 2934, 1605, 1508, 1443, 1409, 1347, 844 cm⁻¹; ¹H-NMR (500 MHz, CDCl₃): δ = 2.18 (s, 6H), 5.02 (s, 2H), 5.91 (s, 2H), 6.89 (dd, *J* = 8.6, 5.4 Hz, 2H), 7.03 (dd, *J* = 8.6, 5.4 Hz, 2H) ppm; ¹³C-NMR (125 MHz, CDCl₃): δ = 12.8, 46.5, 106.0, 116.0 (d, *J* = 21.1 Hz),

TABLE I
Optimization of Reaction Conditions and Comparison of the Reaction of Aniline with 2,5-Hexanedione Catalyzed by PEG-SO₃H with the Other Catalysts Used for this Reaction^a

Entry	Catalyst (mol %)	Solvent	Time (h)	Tem. (°C)	Yield (%)	Reference
1	None	H ₂ O	24	r.t.	10	This work
2	Triton X 100 (2.5)	H ₂ O	24	r.t.	40	This work
3	CTAB (2.5)	H ₂ O	24	r.t.	30	This work
4	SDS (2.5)	H ₂ O	24	r.t.	50	This work
5	SDS (2.5)	H ₂ O	12	80	70	This work
6	LiDS (2.5)	H ₂ O	24	r.t.	55	This work
7	PEG-SO ₃ H (3)	H ₂ O	7	r.t.	85	This work
8	PEG-SO ₃ H (2)	H ₂ O	7	r.t.	87	This work
9	PEG-SO ₃ H (1)	H ₂ O	8	r.t.	92	This work
10	Bi(NO ₃) ₃ (5)	CH ₂ Cl ₂	10	–	96	38
11	Monmorinolute-K10 (3 g)	CH ₂ Cl ₂	26	r.t.	96	37
12	Monmorinolute-KSF (1 g)	–	10	r.t.	95	34
13	Zr(KPO ₄) ₂ (12)	–	24	60	56	24
14	Ionic liquid (1.5 g)	–	3	r.t.	96	40

^a The ratio of aniline/2,5-hexanedione is 2/2.4.

TABLE II
PEG-SO₃H Catalyzed Synthesis of Pyrroles in Water at Room Temperature^a

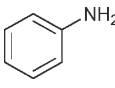
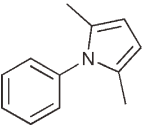
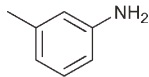
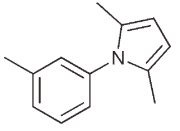
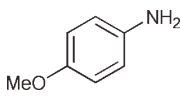
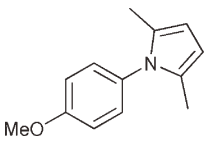
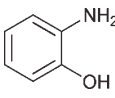
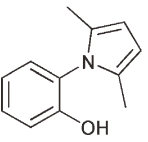
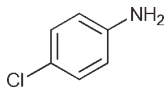
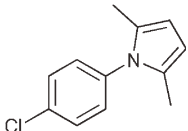
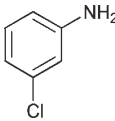
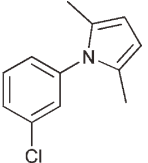
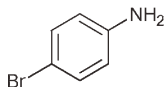
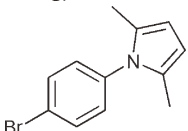
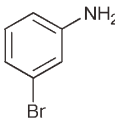
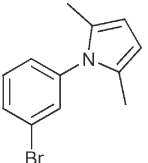
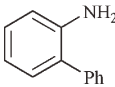
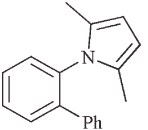
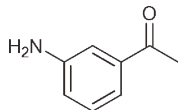
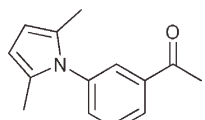
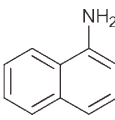
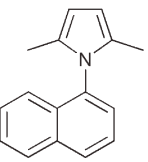
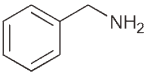
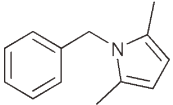
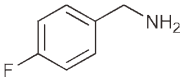
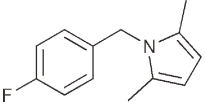
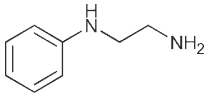
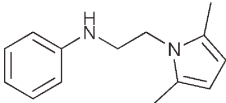
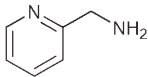
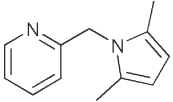
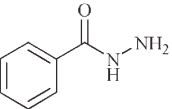
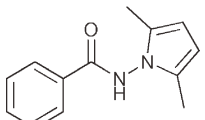
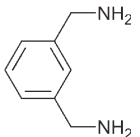
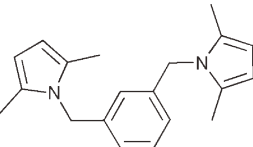
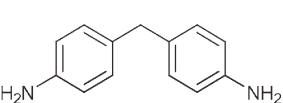
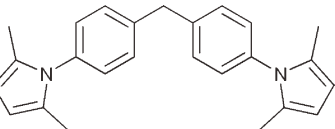
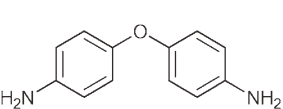
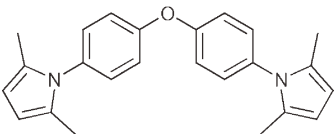
Entry	Amine	Product	Time (h)	Yield (%) ^b	m.p. (reported)	Ref.
1			8	92	49–50 (50)	[34]
2			11	93	50–51	[35]
3			10	95	58–59 (59–60)	[24]
4			10	90	95–97	[31]
5			12	87	47–48	[35]
6			13	90	47–49	[37]
7			15	92	70–72	[35]
8			13	98	83–84	[35]
9			30	88	96–98	[36]
10			20	93	63–65	–
11			24	90	118–119 (118)	[33]

TABLE II. Continued

Entry	Amine	Product	Time (h)	Yield (%) ^b	m.p. (reported)	Ref.
12			2	90	42–43 (42–43)	[24]
13			4	93	55–56	–
14			6	91	68–70	–
15			4	90	53–54	–
16			10	95	181–182	–
17 ^c			2	95	89–91	–
18 ^c			9	93	117–119	–
19 ^c			24	90	170–172	–

^a The reaction conditions: amine (2 mmol), 2,5-hexanedione (2.4 mmol), PEG-SO₃H (0.01 mmol), at room temperature.

^b Yields refer to isolated pure products which were characterized by comparison of their m.p., IR, ¹H and ¹³C NMR spectra with those of authentic samples.

^c The reaction conditions: diamine (2 mmol), 2,5-hexanedione (5 mmol), PEG-SO₃H (0.01 mmol), at room temperature.

127.6 (d, *J* = 8.1 Hz), 128.3, 134.6 (d, *J* = 3.1 Hz), 162.3 (d, *J* = 243.8 Hz) ppm.

N-(2-(2,5-Dimethyl-1*H*-pyrrol-1-yl)ethyl)benzenamine (Table II, entry 14): white solid; m.p.: 69–71°C; FTIR: ν_{\max} = 3354, 2913, 1600, 1509, 1444, 1350, 1325, 773, 750 cm⁻¹; ¹H-NMR (500 MHz, CDCl₃): δ = 2.27 (s, 6H), 3.45 (t, *J* = 6.6 Hz, 2H), 3.80 (s, NH), 4.02 (t, *J* = 6.6 Hz, 2H), 5.86 (s, 2H), 6.65 (d, *J* = 7.7 Hz, 2H), 6.78 (t, *J* = 7.4 Hz, 1H), 7.24 (dd, *J* = 8.3, 7.4 Hz, 2H) ppm; ¹³C-NMR (125 MHz, CDCl₃): δ = 13.0, 43.2, 44.3, 106.1, 113.1, 118.3, 128.2, 129.9, 147.8 ppm.

2-((2,5-Dimethyl-1*H*-pyrrol-1-yl)methyl)pyridine (Table II, entry 15): white solid, m.p.: 53–55°C; FTIR: ν_{\max} = 3040, 2932, 1633, 1591, 1571, 1440, 1350, 768, 751 cm⁻¹; ¹H-NMR (500 MHz, CDCl₃): δ = 2.18 (s, 6H), 5.16 (s, 2H), 5.91 (s, 2H), 6.47 (d, *J* = 7.8 Hz, 1H), 7.19 (dd, *J* = 7.1, 5.0 Hz, 1H), 7.61 (ddd, *J* = 7.8, 7.1, 1.7 Hz, 1H), 8.60 (d, *J* = 5.0 Hz, 1H) ppm; ¹³C-NMR (125 MHz, CDCl₃): δ = 12.8, 49.3, 106.2, 120.3, 122.5, 128.4, 137.7, 149.7, 159.0 ppm.

N-(2,5-dimethyl-1*H*-pyrrol-1-yl)benzamide (Table II, entry 16): white solid; m.p.: 180–182°C; FTIR: ν_{\max} = 3160, 2988, 1695, 1660, 1577, 1485, 1326, 787, 744

TABLE III
Reusability of PEG-SO₃H in the Synthesis of
1-(4-Fluorobenzyl)-2,5-dimethyl-1H-Pyrrole at
Room Temperature^a

Run	1	2	3	4
Yield (%)	93	92	92	93
Time (h)	4	4	4.25	4.25

^aThe ratio of aniline/2,5-hexanedione is 2/2.4.

cm⁻¹; ¹H-NMR (500 MHz, CDCl₃): δ = 2.14 (s, 6H), 5.88 (s, 2H), 7.51 (t, *J* = 7.6 Hz, 2H), 7.62 (t, *J* = 7.3 Hz, 1H), 7.85 (d, *J* = 7.6 Hz, 2H), 8.62 (s, 1H, NH) ppm; ¹³C-NMR (125 MHz, CDCl₃): δ = 11.6, 104.5, 127.8, 128.5, 129.3, 132.1, 133.0 ppm.

1-(3-((2,5-Dimethyl-1H-pyrrol-1-yl)methyl)benzyl)2,5-dimethyl-1H-pyrrole (Table II, entry 17): white solid, m.p.: 89–91°C; FTIR: ν_{\max} = 3000, 2930, 1607, 1520, 1443, 1407, 1325, 1301, 974, 751 cm⁻¹; ¹H-NMR (500 MHz, CDCl₃): δ = 2.16 (s, 12H), 5.00 (s, 4H), 5.90 (s, 4H), 6.59 (s, 1H), 6.74 (d, *J* = 7.5 Hz, 2H), 7.24 (t, *J* = 7.5 Hz, 1H) ppm; ¹³C-NMR (125 MHz, CDCl₃): δ = 12.9, 47.1, 105.9, 123.6, 124.9, 128.3, 129.7, 139.6 ppm.

Bis(4-(2,5-dimethyl-1H-pyrrol-1-yl)phenyl)methane (Table II, entry 18): pale yellow solid; m.p.: 117–119°C; FTIR: ν_{\max} = 2920, 1516, 1402, 1320, 998, 822, 744 cm⁻¹; ¹H-NMR (400 MHz, CDCl₃): 2.06 (s, 12H), 4.12 (s, 2H), 5.91 (s, 4H), 7.18 (d, *J* = 8.0 Hz, 4H), 7.31 (d, *J* = 8.0 Hz, 4H); ¹³C-NMR (100 MHz, CDCl₃): δ = 13.2, 41.1, 105.7, 128.4, 128.9, 139.7, 137.3, 140.0 ppm.

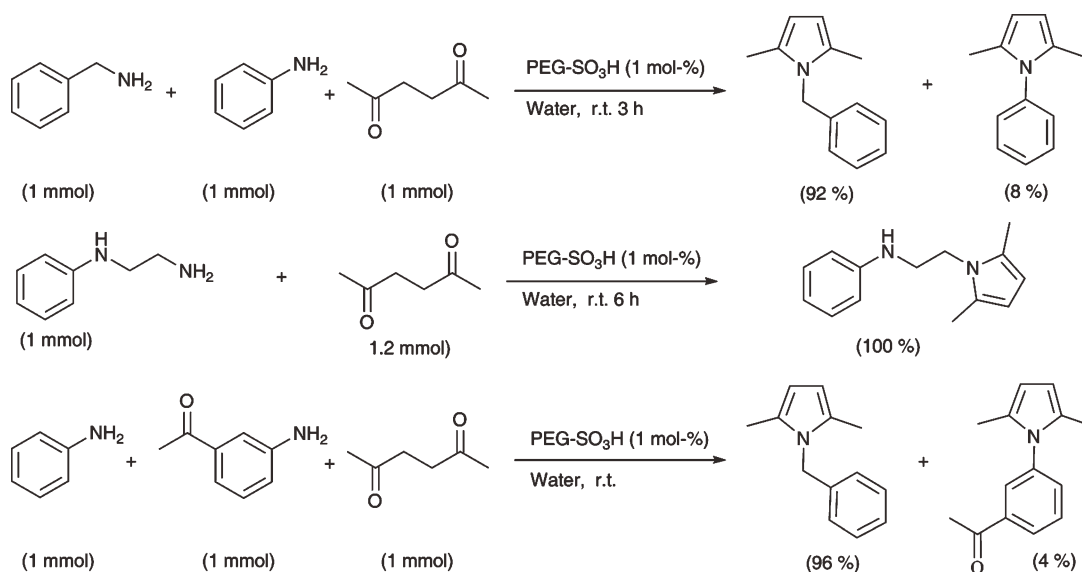
1,1'-(4,4'-Oxybis(4,1-phenylene))bis(2,5-dimethyl-1H-pyrrole) (Table II, entry 19): pale yellow solid, m.p.: 170–172°C, FTIR: ν_{\max} = 2919, 1501, 1403, 1234, 998, 839, 757, 650 cm⁻¹; ¹H-NMR (400 MHz, CDCl₃): 2.07

(s, 12H), 5.92 (s, 4H), 7.15 (d, *J* = 8.8 Hz, 4H), 7.23 (d, *J* = 8.8 Hz, 4H) ppm; ¹³C-NMR (100 MHz, CDCl₃): δ = 13.1, 105.7, 119.3, 128.9, 129.7, 134.5, 156.2 ppm.

RESULTS AND DISCUSSION

At first, for our initial optimization studies, condensation of aniline with 2,5-hexanedione was chosen as a model reaction. Thus, a mixture of aniline (2 mmol) and 2,5-hexanedione (2.2 mmol) in water was stirred in the presence of some surfactant catalysts at room temperature (Table I). The reaction in the presence of triton X-100, cetyl trimethylammonium bromide (CTAB), sodium dodecyl sulfate (SDS), and lithium dodecyl sulfate (LiDS) did not proceed to completion even after 24 h (Table I, entries 2–6). We observed a drastic rate enhancement when using PEG-SO₃H as a catalyst to produce the desired pyrrole in 92% isolated yield (Table I, entry 9). Next, the amount of the catalyst was optimized, and 1 mol % of PEG-SO₃H was chosen as an optimal amount (Table I, entries 7–9). As shown in the head of Table I, the product was precipitated and isolated after the completion of the reaction and the addition of water (4 mL) to the reaction mixture. The precipitated product was filtered off and washed with water to yield pure 2,5-dimethyl-1-phenyl-1H-pyrrole in 92% yield. This simple workup opened the route for an entirely green and highly efficient Paal-Knorr reaction in water.

In addition, the efficacy of PEG-SO₃H can be gauged by comparing with other catalysts used in similar reactions (Table I, entries 10–14). It is worth mentioning that our method is more efficient and simpler than most of the existing methods. More



Scheme 2 Competitive reaction in Paal-Knorr pyrrole synthesis catalyzed by PEG-SO₃H in water at room temperature.

importantly, it uses no organic solvents in reaction media and/or work-up.

The above results prompted us to study the generality and the scope of PEG-SO₃H-catalyzed synthesis of pyrroles. Thus, various amines were condensed with 2,5-hexanedione to provide the desired pyrroles. All the aforementioned reactions proceeded well enough and delivered high yields with a range of aliphatic and aromatic amines containing electron-donating and electron-withdrawing groups (Table II). In all the cases, the pure product was isolated by a simple filtration without using any chromatography or cumbersome reaction workup.

The 3-pyrrolyl acetophenone (Table II, entry 10), pyrrolyl ethylbenzenamine (Table II, entry 14), pyrrolyl methylpyridine (Table II, entry 15), and benzamido pyrrole (Table II, entry 16) were produced as new products under the same conditions to show the selectivity and the scope of this green procedure. The corresponding pyrrole derivatives were formed obviously in excellent yields and purity.

Bispyrroles are interesting compounds. For example, they can be employed as the monomers for the preparation of macrocyclic or polymeric compounds with pyrrole structures. Through this study, investigation was also made of the applicability of the method for the preparation of bispyrrole via the reaction of structurally diverse diamines, and 2,5-hexanedione in the presence of 1 mol % of PEG-SO₃H in water at room temperature. The desired bispyrroles were precipitated as the reaction proceeded. The sole products were achieved in high yields after addition of water to the reaction mixture and isolated by a simple filtration (Table II, entries 17–19).

The reusability of the catalysts is one of the most important benefits which makes them useful for commercial applications. Therefore, we investigated the recovery and reusability of PEG-SO₃H in the condensation of *p*-fluorobenzyl amine with 2,5-hexanedione. As it was mentioned, the solid product can be easily separated by a simple filtration from the reaction mixture. The reaction medium (solution of PEG-SO₃H in water) was used after reducing the volume of water. As it can be seen (Table III), the catalyst could be used at least for four successive times without any reduction in its activity.

Selectivity of the method is important for the total synthesis of complex organic molecules. To show the high selectivity of the method, we performed three competitive reactions for the Paal–Knorr pyrrole synthesis using PEG-SO₃H as catalyst in water at room temperature (Scheme 2). The highly selective conversion of aliphatic amine was observed *versus* aromatic ones. Similarly, the competitive reactions of aniline *versus* substituted aniline with electron-withdrawing carbonyl group led to the exclusive pyrrole product of aniline. The results

obtained show the importance of the electronic effects and nucleophilicity of nitrogen lone pair upon the PEG-SO₃H catalyzed reaction.

CONCLUSIONS

In conclusion, we have developed an environmentally benign method for the condensation of amines with 2,5-hexanedione at room temperature, avoiding the use of any organic solvent in reaction media and/or in work-up. The simple experimental and product isolation procedures combined with the ease of recovery and reuse of the reaction medium is expected to contribute to the development of a green strategy for the preparation of pyrroles via Paal–Knorr reaction.

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