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Short communication

Intramolecular imino-Diels–Alder reactions in [bmim]BF_4 ionic medium: Green protocol for the synthesis of tetrahydrochromanoquinolines

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Abstract

2-Azadienes derived *in situ* from aryl amines and *O*-prenyl derivatives of salicylaldehydes under intramolecular [4 + 2] imino-Diels–Alder reaction, in an air and moisture stable [bmim]BF_4 ionic medium afford tetrahydrochromanoquinolines in high to quantitative yield. This new protocol offers significant advantages over reported methods which include mild reaction conditions, high conversions, enhanced reaction rates and recyclability of ionic liquids.

Keywords: Imino-Diels–Alder; Tetrahydrochromanoquinolines; *O*-Prenyl derivative of aryl aldehyde; Aryl amine; Ionic liquids (ILs)

1. Introduction

The Aza-Diels–Alder reaction is becoming a mainstay for heterocycle and natural product synthesis [1]. Tetrahydroquinolines are a very important class of compounds in the filed of drugs and pharmaceuticals as psychotropic, antiallergenic, anti-inflammatory and estrogenic agents [2]. The imino-Diels–Alder reaction is a useful synthetic tool for constructing *N*-containing six-membered heterocycles such as tetrahydroquinolines, octohydroacridines, tetrahydrochromanoquinoles and dihydro-4-pyridones [3,4]. Imines derived from aromatic amines act as heterodienes and undergo imino-Diels–Alder reactions with electron rich dienophiles [1]. The most straightforward method for the synthesis of tetrahydrochromanoquinolines involves an acid catalysed intramolecular cyclisation of aryl amines with non-activated olefins tethered to the diene system [5]. Acid catalyst such as $Yb(OTf)_3$, TFA, $BiCl_3$, $LiClO_4$, and TPP·$HClO_4$ have been reported to accomplish this reaction [5,6]. However, most of these procedures involve the use of corrosive and hazardous reagent which always demand a routine aqueous work-up for the catalyst separation, recycling and disposal.

Ionic liquids (ILs) have recently emerged as recyclable reaction media for the immobilisation of transition metal-based catalysts, Lewis acids and enzymes [7]. They are being used as a set of green solvents with unique properties such as tenable polarity, high thermal stability, immiscibility with a number of organic solvents, negligible vapour pressure and ease of recyclability. They are referred to as 'designer solvents' as their properties such as hydrophilicity, hydrophobicity, Lewis acidity, viscosity and density can be altered by the fine-tuning of parameters such as the choice of organic cation, inorganic anion and the length of alkyl chain attached to an organic cation (Fig. 1) [8].

These structural variations offer flexibility to the chemist to devise the optimal solvent, catering to the needs of any particular process. Since ionic liquids are entirely composed of ions, they provide an ideal reaction medium for reactions that involve reactive ionic intermediates. Due to the stabilization of charged intermediates by ionic liquids, they can promote enhanced selectivity and reaction rates. As a result of their green credentials and potential to enhance reaction rates and selectivity, ionic liquids are finding increasing applications in organic synthesis [9]. Fur-

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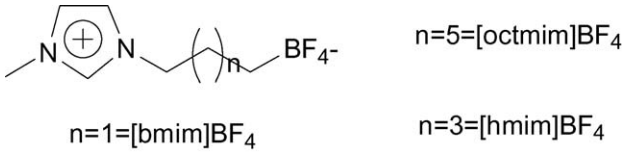


Fig. 1. Chemical structure of ionic liquid.

thermore, ionic liquids act as efficient dehydrating media for imine formation [10].

2. Experimental

2.1. General methods

Melting points were recorded on a Buchi R-535 apparatus and are uncorrected. IR spectra were recorded on a Perkin-Elmer FT-IR 240-c spectrophotometer using KBr optics. ^{1}H NMR and ^{13}C spectra were recorded on Gemini-200 spectrometer (200 MHz) in $CDCl_3$ using TMS as internal standard. Mass spectra were recorded on a Finnigan MAT 1020 mass spectrometer operating at 70 eV. Column chromatography was performed using E. Merck 60–120, mesh silica gel. All solvents were distilled, dried over CaH_2 and stored under nitrogen prior to use. Starting materials and reagents used in the reactions were obtained commercially from Aldrich, Lancaster, Fluka and were used without purification, unless otherwise indicated.

2.2. General procedure

A mixture of *O*-prenyl derivative of aryl aldehyde (1 mmol), aryl amine (1 mmol) and ionic liquid [bmim]BF_4 (3 mL) was stirred at room temperature for the appropriate time (Table 1). After completion of the reaction as indicated by TLC. The reaction mixture was extracted with diethyl ether (3× 10 mL). The combined organic layers were dried over anhydrous Na_2SO_4, concentrated in vacuum and purified by column chromatography on silica gel (ethyl acetate–hexane, 0.5–9.5) to afford pure chromanoquinoline. The combined organic layer was evaporated under reduced pressure to afford a mixture of *cis* and *trans* tetrahydrochromano [4,3-*b*] quinolines. The remaining ionic liquid was further washed with ether and recycled in subsequent reactions.

2.2.1. Spectral data for new products

2.2.1.1. 5j. Yellow crystalline solid; m.p. 109–110 °C; IR (KBr): ν_{max}: 3377, 2924, 1602, 1487, 1230, 744 cm^{-1}; ^{1}H NMR ($CDCl_3$, 200 MHz): δ 1.62 (s, 3H, CH_3), 1.68 (s, 3H, CH_3), 2.14 (td, 1H, H-8a, J = 10.3, 3.2 Hz), 3.96 (t, 1H, H-8′, J = 10.6 Hz), 4.23 (brs, 1H, NH), 4.43 (dd, 1H, H-8, J = 3.1, 10.0 Hz), 5.29 (d, 1H, H-14a, J = 10.4 Hz), 6.37–8.25 (m, 10H, Ar); ^{13}C NMR ($CDCl_3$, 50 MHz), δ 151.5, 140.4, 132.6, 129.6, 129.1, 128.8, 127.3, 127.2, 126.6,125.5, 123.4, 121.4, 118.9, 117.0, 115.0, 113.4, 63.6, 42.3, 40.2, 33.5, 33.4, 25.6; EI-MS: *m*/*z* (%) 315 (*M*+), 300 (17), 271 (22), 182 (100), 152 (33), 132 (15), 117 (17), 41 (10).

2.2.1.2. 5k. Yellow crystalline solid; m.p. 58–60 °C; IR (KBr): ν_{max}: 3370, 2450, 1505, 1235, 757 cm^{-1}; ^{1}H NMR ($CDCl_3$, 200 MHz): δ 1.29 (s, 3H, CH_3), 1.45 (s, 3H, CH_3), 1.95 (td, 1H, H-8a, J = 10.3, 3.0 Hz), 2.18 (s, 3H, Ar–CH_3), 3.76 (t, 1H, H-8′, J = 10.7 Hz), 3.92 (brs, 1H, NH), 4.25 (dd, 1H, H-8, J = 3.3, 11.0 Hz), 5.10 (d, 1H, H-14a, J = 9.8 Hz), 6.80–8.08 (m, 9H, Ar); ^{13}C NMR ($CDCl_3$, 50 MHz), δ 153.4, 138.0, 132.5, 129.4, 129.0, 128.7, 127.8, 127.0, 126.6, 125.9, 123.3, 121.4, 118.8, 115.2, 113.4, 77.5, 76.9, 76.3, 33.5, 33.3, 28.7, 25.6, 20.6; EI-MS: *m*/*z* (%) 329 (*M*+), 327 (73), 314 (47), 284 (20),194 (12), 181 (57), 157 (20), 148 (22), 106 (18), 41 (17).

2.2.1.3. 5l. Yellow crystalline solid; m.p. 194–196 °C; IR (KBr): ν_{max}: 3366, 2960, 1596, 1485, 1227, 1085, 808, 742 cm^{-1}; ^{1}H NMR ($CDCl_3$, 200 MHz): δ 1.48 (s, 3H, CH_3), 1.52 (s, 3H, CH_3), 2.05 (td, 1H, H-8a, J = 10.3, 2.3 Hz), 3.75 (t, 1H, H-8′, J = 11.0 Hz), 4.15 (brs, 1H, NH), 4.22 (dd, 1H, H-8, J = 3.3, 11.0 Hz), 5.14 (d, 1H, H-14a, J = 9.2 Hz), 6.20–8.09 (m, 9H, Ar); ^{13}C NMR ($CDCl_3$, 50 MHz), δ 151.3, 139.4, 132.3, 129.9, 129.7, 129.4, 128.8, 128.1, 127.2, 125.9, 123.4, 121.1, 118.8, 114.8, 113.4, 63.2, 42.2, 40.3, 39.6, 33.5, 33.0, 25.3; EI-MS: *m*/*z* (%) 394 (*M*+), 348 (10), 328 (23), 238 (09), 222 (12), 181 (100), 171 (38), 144 (18), 115 (18), 69 (20), 41 (20).

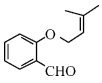
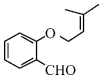
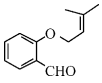
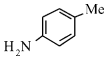
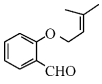
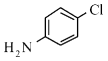
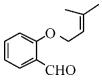
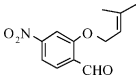
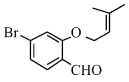
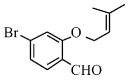
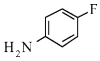
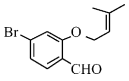
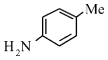
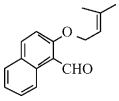
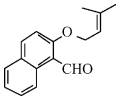
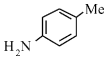
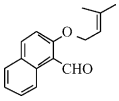
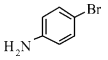
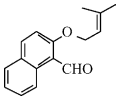
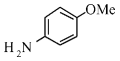
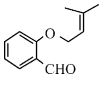
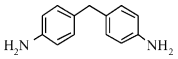
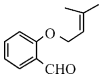
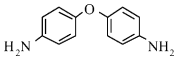
2.2.1.4. 5m. Yellow crystalline solid; m.p. 102–104 °C; IR (KBr): ν_{max}: 3397, 2970, 2832, 2358, 1622, 1504, 1291, 1169, 814, 768 cm^{-1}; ^{1}H NMR ($CDCl_3$, 200 MHz): δ 1.50 (s, 3H, CH_3), 1.56 (s, 3H, CH_3), 2.05 (td, 1H, H-8a, J = 10.4, 3.3 Hz), 3.78 (s, 3H, –OCH_3), 3.90 (t, 1H, H-8′, J = 11.3 Hz), 4.32 (dd, 1H, H-8, J = 3.0, 11.1 Hz), 5.13 (d, 1H, H-14a, J = 11.3 Hz), 5.30 (brs, 1H, NH), 6.28–8.15 (m, 9H, Ar); ^{13}C NMR ($CDCl_3$, 50 MHz), δ 151.7, 151.4, 134.5, 132.5, 129.5, 129.1, 128.7, 128.1, 127.1, 123.4, 121.5, 118.8, 115.2, 114.3, 112.7, 111.9, 96.1, 63.5, 42.4, 40.2, 33.6, 33.5, 25.7; EI-MS: *m*/*z* (%) 345 (*M*+), 332 (25), 213 (58), 182 (30),158 (65), 129 (22), 118 (62), 69 (15), 41 (23).

2.2.1.5. 10a. White crystalline solid; m.p. 112–115 °C; IR (KBr): ν_{max}: 2924, 1602, 1458, 1031, 744 cm^{-1}; ^{1}H NMR ($CDCl_3$, 200 MHz): δ 5.39 (s, 2H, –OCH_2), 6.96 (d, 1H, J = 8.3 Hz), 7.13–7.18 (m, 1H), 7.22–7.42 (m, 1H), 7.42–7.56 (m, 1H), 7.65–7.79 (m, 2H), 7.82 (s, 1H), 8.09 (d, 1H, J = 7.9 Hz), 8.47 (d, 1H, J = 5.8 Hz); EI-MS: *m*/*z* (%) 233 (*M*+), 204 (19), 141 (20), 105 (18), 91 (15), 77 (20), 57 (30), 43 (52).

2.2.1.6. 10b, 10e. Yellow crystalline solid; m.p. 91–93 °C; IR (KBr): ν_{max}: 2924, 1591, 1464, 1219, 1035, 763 cm^{-1}; ^{1}H NMR ($CDCl_3$, 200 MHz): δ 2.63 (s, 3H, Ar–CH_3), 5.45 (s, 2H, –OCH_2), 6.94 (d, 1H, J = 8.2 Hz), 7.05–7.12 (m, 1H), 7.28–7.35 (m, 1H), 7.40–7.46 (m, 1H), 7.62–7.68 (m, 1H), 7.92 (d, 1H, J = 8.2 Hz), 8.05 (d, 1H, J = 8.0 Hz), 8.46 (d, 1H, J = 7.8 Hz); EI-MS: *m*/*z* (%) 247 (*M*+), 233 (47), 128 (29), 99 (23), 71 (47), 57 (100), 47 (52).

2.2.1.7. 10c. Yellow crystalline solid; m.p. 144–146 °C; IR (KBr): ν_{max}: 2923, 2855, 1584, 1458, 1215, 1001, 756 cm^{-1}; ^{1}H NMR ($CDCl_3$, 200 MHz): δ 2.58(s, 6H, Ar–CH_3), 5.38 (s, 2H,

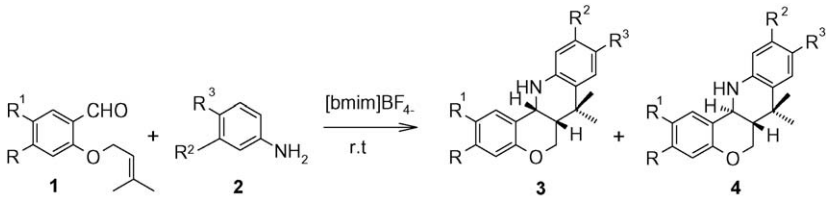
Table 1
[bmim]BF_4-promoted synthesis of tetrahydrochromanoquinolines

Entry	Salicylaldehyde	Aniline	Product[a]	Time (h)	Yield (%)[b]
a			3a/4a	4.0	93
b			3b/4b	3.5	90
c			3c/4c	4.0	88
d			3d/4d	5.5	86
e			3e/4e	6.5	85
f			3f/4f	7.5	75
g			3g/4g	6.0	81
h			3h/4h	5.5	78
I			3i/4i	4.5	85
j			5j	3.5	94
k			5k	3.0	92
l			5l	3.5	88
m			5m	2.5	92
n			6n/7n/8n	6.5	79
o			9o	7.5	75

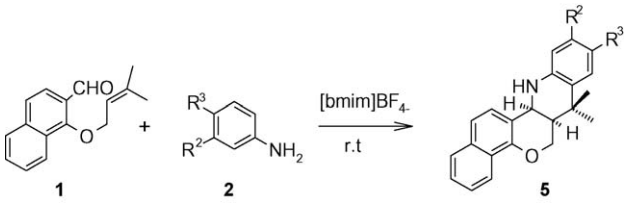
[a] All products were characterised by ^{1}H NMR and IR spectroscopy and mass spectrometry.
[b] Isolated and unoptimised yields.

–OCH_2), 6.92 (d, 1H, J = 8.0 Hz), 7.02–7.12 (m, 1H), 7.22–7.32 (m, 1H), 7.43 (d, 1H, J = 8.6 Hz), 7.63 (s, 1H) 7.96 (d, 1H, J = 8.7 Hz), 8.46 (d, 1H, J = 7 Hz); EI-MS: m/z (%) 261 (M+), 246 (29), 155 (08), 141 (29), 100 (08), 99 (08), 85 (47), 71 (68), 57 (100), 43 (93).

2.2.1.8. 10d. Yellow liquid; IR (KBr): ν_{max}: 3465, 2963, 2362, 1218, 770 cm^{-1}; ^{1}H NMR ($CDCl_3$, 200 MHz): δ 2.60(s, 3H, Ar–CH_3), 5.46 (s, 2H, –OCH_2), 6.92 (d, 1H, J = 8.3 Hz), 7.08–7.14 (m, 1H), 7.30 (m, 5H), 7.52 (m, 1H), 7.63 (s, 1H) 7.70 (m, 1H) 8.00 (d, 1H, J = 8.3 Hz), 8.46 (d, 1H, J = 5.9 Hz);



Scheme 1.



Scheme 2.

EI-MS: *m*/*z* (%) 323 (*M*+), 247 (47), 141 (36), 83 (100), 57 (10), 47 (48).

3. Results and discussion

In this article, we describe ionic liquids (ILs) as recyclable reaction media for the intramolecular [4 + 2] cycloaddition of aryl amines with *O*-prenyl derivatives of salicylaldehydes under neutral conditions (Scheme 1).

The reactions proceeded smoothly in ionic liquid without the need for any additional acid catalyst. In this reaction, ionic liquids play a dual role as solvents and catalyst. For example, treatment of aniline with the *O*-prenyl derivative of salicylaldehyde in the ionic liquid [bmim]BF_4 at RT, afforded 7,7-dimethyl-6a,7,12,12a-tetrahydro-6*H*-chromeno[4,3-b] quinoline in 93% yield (entry a, Table 1). Similarly, various aromatic imines underwent smooth intermolecular imino-Diels–Alder reaction to provide the corresponding tetrahydrochromanoquinolines. In most cases, the product was obtained as a mixture of *cis*-3 and *trans*-isomers 4 in approximately 1:1 ratio. The diasteromers could be easily separated by simple flash chromatography. The stereochemistry of the products was assigned on the basis of coupling constants of the protons in the ^{1}H NMR spectra and also by direct comparison with authentic samples [6]. The diastereomeric ratio was determined from the NMR spectra of the crude products. In the case of 2-hydroxy-1-naphtaldehyde, the product was obtained exclusively as a *cis*-isomer because of steric effect naphthalene (entries 5j–m, Table 1) (Scheme 2).

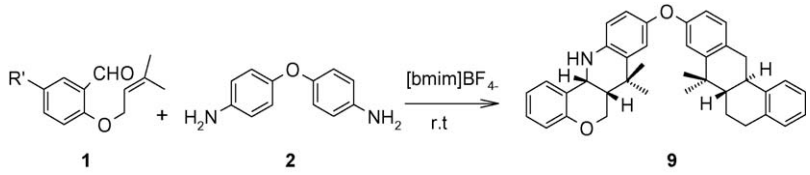
Table 2
Comparative study of various solvents and catalysts for the condensation of aniline with *O*-prenyl salicylaldehyde[a]

Entry	Solvent	Catalyst	Time (h)	Yield (%)
a	[bmim]BF_4 (3 mL)	No catalyst	4.0	93
b	[hmim]BF_4 (3 mL)	No catalyst	4.5	89
c	[octmim]BF_4 (3 mL)	No catalyst	5.0	85
d	[bmim]BF_6 (3 mL)	No catalyst	8.0	65
e	CH_3CN	5 mol% $Yb(OTf)_3$ or 1% TFA	0.5	68
f	Diethyl ether	5 MLPDE	6.5	85
g	CH_3CN	20 mol% $PPh_3{\cdot}HClO_4$	1.5	81
h	CH_3CN	10 mol% $BiCl_3$	1.0	83

[a] Aniline: *O*-prenyl salicylaldehyde = 1:1; 1 mmol of each compound used.

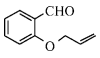
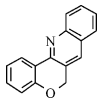
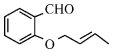
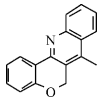
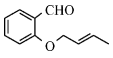
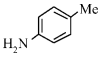
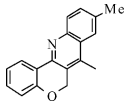
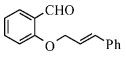
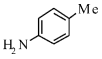
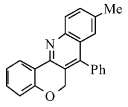
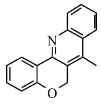
The exclusive formation of *cis*-isomer in the reactions between the *O*-prenyl derivative of 2-naphthal and aryl amines is presumably due to the steric effect of the naphthyl ring. In further experiments, aromatic diamines having two amino groups on the different aromatic rings were studied. Treatment of 4,4′-methylenedianiline with the *O*-prenyl derivative of salicylaldehye in [bmim]BF_4 afforded the biscyclisation product as mixture of *cis/cis* 6, *cis/trans* 7, *trans/trans* 8. These three products could be easily separated and characterised by ^{1}H NMR, IR and mass spectral analysis. In the case of 4,4′-oxydianiline, the product was obtained exclusively as *cis*-, *trans*-, bis-adduct 9 under similar conditions (Scheme 3, Table 1). The scope and generality of this method is illustrated with respect to various *O*-hydroxybenzaldehyes and a wide range of aryl amines and the results are presented in Table 1.

In the absence of solvent (ionic liquid), the reaction did not proceed even after a along reaction time (8–12 h). In this reaction, the efficiency of the ionic liquid was strongly influenced by the nature of the anion. The reactivity of aryl amines with the *O*-prenyl derivative of salicylaldehyde was studied in both hydrophilic [bmim]BF_4 and hydrophobic [bmim]BF_6 ionic liquids. Of these 1-butyl-3-methylimidazolium tetrafluoroborate ([bmim]BF_4) was found to be the most effective in terms of conversion and reaction rates. Aryl imines, formed



Scheme 3.

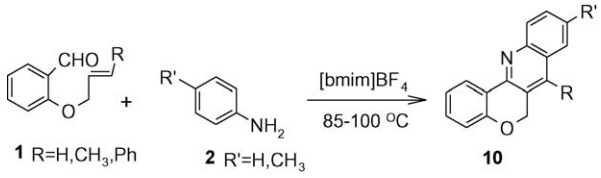
Table 3
[bmim]BF_4-promoted synthesis of chromanoquinolines

Entry	Salicylaldehyde 1	Aniline 2	Product 10	Temperature (°C)/time (h)	Yield (%)[a]
a				100/24	56
b				85/16	75
c				85/14	80
d				90/22	65
e				85/16	71

All products were characterised by ^{1}H NMR and IR spectroscopy and mass spectrometry.
[a] Isolated and unoptimised yields.

in situ form anilines and *O*-prenyl derivative of salicylaldehydes, exhibit enhanced reactivity in ionic liquid thereby reducing the reaction times and improving the yields significantly (see Table 2) for instance treatment of *O*-prenyl ether of salicylaldehyde with aniline in [bmim]BF_4 afforded the corresponding chromanoquinoline 3a/4a in 93% yield whereas the same reaction in the absence of acid catalyst, in commonly used organic solvents such as acetonitrile or methanol gave the intermediate imine after 3 h with no further cyclisation. In addition the ionic liquid was easily recovered after the reaction and reused in subsequent reactions. The recovered ionic liquid was reused for 3–5 times without loss of activity. Even after the fourth cycle the product 3a/4a was obtained with a similar yield and purity as that obtained in the first cycle. The commercially available Fluka ionic liquids were used for this study. The purity of [bmim]BF_4 ionic liquid $\geq$ 97% (NMR). The use of ionic liquids as the reaction media of this transformation helps to avoid the necessity of moisture sensitive reactant for heavy metal Lewis acids as promoters thereby minimizing the production of toxic waste during work-up. Similar results were also obtained with ionic liquids having longer alkyl chain such as 1-hexyl-3-methylimidazolium tetrafluoroborate [hmim]BF_4 or 1-octyl-3-methylimidazolium tetrafluoroborate[octmim]BF_4 (see Table 2). Further we have studied the condensation of aryl amines with less reactive substrates such as 2-allyloxybenzaldehyde, 2-[(E)-2-butenyloxy]benzaldehyde, 2-[(Z)-2-butenyloxy]benzaldehyde, 2-[3phenyl-(E)-2-propenyloxy] benzaldehyde using [bmim]BF_4 ionic media. Interestingly, these substrates gave a new class of chromanoquinolines, instead of the expected tetrahydrochromanoquinolines (Scheme 4).

However, these reactions proceeded only under heating conditions and the results are presented in Table 3.



Scheme 4.

4. Conclusion

In conclusion, we describe a novel convenient and one-pot procedure for the synthesis of chromanoquinolines through an intramolecular imino-Diels–Alder reaction of *N*-arylimines derived from aromatic amines and *O*-prenyl derivative of salicylaldehydes using air- and moisture-stable ionic liquid as a recyclable reaction medium. The ionic liquid plays a dual role as solvent and the promoter in this conversion. The arylimines exhibit enhanced reactivity in ionic liquids thereby reducing the reaction times and improving the yield significantly. The simple experimental and product isolation procedures combined with ease of recovery and reuse of this novel reaction media is expected to contribute to the development of a green strategy for the synthesis of highly fictionalized chromanoquinolines.

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