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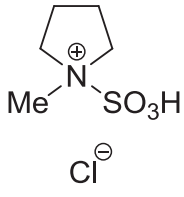
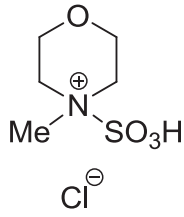
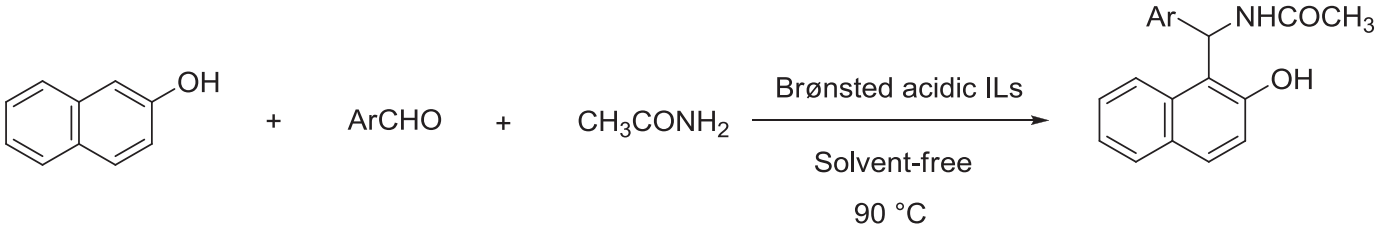


[MPyrrSO$_3$H]Cl (**IL$_1$**)



[MMorSO$_3$H]Cl (**IL$_2$**)



ORGANIC CHEMISTRY | RESEARCH ARTICLE

Another application of newly prepared Brønsted-acidic ionic liquids as highly efficient reusable catalysts for neat synthesis of amidoalkyl naphthols

Maryam Dehghan, Abolghasem Davoodnia, Mohammad R. Bozorgmehr and Niloofar Tavakoli-Hoseini

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Another application of newly prepared Brønsted-acidic ionic liquids as highly efficient reusable catalysts for neat synthesis of amidoalkyl naphthols

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Abstract: In this work, two newly prepared Brønsted-acidic ionic liquids, [MPyrrSO$_3$H]Cl (**IL$_1$**) and [MMorSO$_3$H]Cl (**IL$_2$**), were efficiently used as catalysts for the synthesis of amidoalkyl naphthols through the one-pot, three-component reaction of β-naphthol, aryl aldehydes, and acetamide under neat conditions. High activity of the catalysts, excellent yields, short reaction times, simple procedure with an easy work-up, and the absence of any volatile and hazardous organic solvents are some advantages of the present methodology. Moreover, the catalysts are simply prepared and can be recovered conveniently and reused such that considerable catalytic activity can still be achieved after the fifth run.

Subjects: Organic Chemistry; Catalysis; Environmental

Keywords: Brønsted-acidic ionic liquids; amidoalkyl naphthols; solvent-free conditions

1. Introduction

A major challenge in modern chemistry is the design of highly efficient chemical reaction sequences that provide maximum structural complexity with a minimum number of synthetic steps in short reaction times (Dömling, 2006; Schreiber, 2000). Multicomponent reactions (MCRs) have gained considerable attention as a powerful method in organic synthesis and medicinal chemistry because they involve simultaneous reaction of more than two starting materials to yield a single product through one-pot reaction (Gore & Rajput, 2013; Slobbe, Ruijter, & Orru, 2012; Tavakoli-Hoseini &

Abolghasem Davoodnia

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Abolghasem Davoodnia was born in 1971, Mashhad, Iran. He studied chemistry at Tehran University, Tehran, Iran, where he received BSc in 1994. He received his MSc degree in organic chemistry in 1997 from Ferdowsi University of Mashhad, Mashhad, Iran, under the supervision of prof Majid M. Heravi and completed his PhD in organic chemistry in 2002 under the supervision of prof Mehdi Bakavoli at the same university. Currently, he is working as a professor at the Chemistry Department, Mashhad Branch, Islamic Azad University, Mashhad, Iran. He has published over 140 peer-reviewed articles in ISI journals. His current research interest is on heterocyclic chemistry, catalysis and new synthetic methodologies.

PUBLIC INTEREST STATEMENT

Application of new ionic Liquids in organic transformations is of great interest in recent years. Therefore, in this paper, two newly prepared Brønsted-acidic ionic liquids were efficiently used as catalysts for the synthesis of amidoalkyl naphthols through the one-pot, three-component reaction of β-naphthol, aryl aldehydes, and acetamide under neat conditions. Some advantages of this procedure are high yields, short reaction times, easy work-up, absence of volatile and hazardous solvents, and reusability of catalysts for a number of times without appreciable loss of activity.

Davoodnia, 2011). High atom economy, good selectivity, time and energy saving, low cost, minimum waste production, and short reaction time make MCRs suitable for the synthesis of complex molecules with potential biological activity (Chebanov & Desenko, 2012; Manjappa, Peng, Jhang, & Yang, 2016; Zang, Zhang, Zang, & Cheng, 2010). On the other hand, the nature of the catalyst plays a crucial role in the determination of the product and selectivity (Khan, Khan, & Bannuru, 2010; Mirzaei & Davoodnia, 2012; Shaterian & Mohammadnia, 2012). Therefore, development of inexpensive, mild, and reusable catalysts for MCRs such as the synthesis of amidoalkyl naphthols remains of interest to the synthetic organic chemists. It has been reported that amidoalkyl naphthols can convert to important biologically active aminoalkyl naphthol derivatives by amide hydrolysis. Later compounds have been evaluated for the hypotensive and bradycardiac effects (Dingermann, Steinhilber, & Folkers, 2004; Shen, Tsai, & Chen, 1999). Amidoalkyl naphthols are generally synthesized via the three-component reaction of β-naphthol, an aldehyde, and an amide in the presence of various catalysts, such as $Sb(OAc)_3$ (Hakimi, 2016), zirconocene dichloride (Cp_2ZrCl_2) (Khanapure, Jagadale, Salunkhe, & Rashinkar, 2016), $ZrOCl_2{\cdot}8H_2O$ (Sheik Mansoor, Aswin, Logaiya, & Sudhan, 2016), nano Al_2O_3 (Kiasat, Hemat-Alian, & Saghanezhad, 2016), carbon-based solid acid (Davoodnia, Mahjoobin, & Tavakoli-Hoseini, 2014), H_3BO_3 (Shahrisa, Esmati, & Nazari, 2012), iodine (Nagawade & Shinde, 2007), nano-sulfated zirconia (Zali & Shokrolahi, 2012), $K_5CoW_{12}O_{40}{\cdot}3H_2O$ (Nagarapu, Baseeruddin, Apuri, & Kantevari, 2007), copper *p*-toluenesulfonate (Wang & Liang, 2011), $Al(H_2PO_4)_3$ (Shaterian, Amirzadeh, Khorami, & Ghashang, 2008), $Yb(OTf)_3$ in [bmim][BF_4] (Kumar, Rao, Ahmad, & Khungar, 2009), and nano silica phosphoric acid (Bamoniri, Mirjalili, & Nazemian, 2014). Although each of these individual methods has its own merits, many suffer from limitations such as long reaction times, unsatisfactory yields, and the use of relatively expensive catalysts. Thus, the exploration of novel methodologies using new efficient and reusable catalysts is still ongoing.

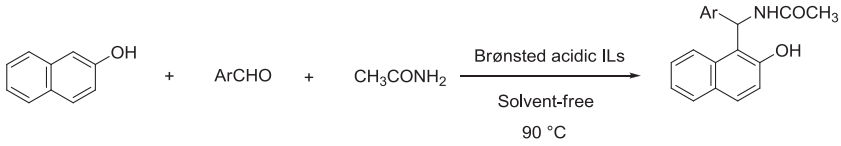
In recent years, ionic Liquids (ILs) have attracted rising interest as eco-friendly solvents, catalysts and reagents in organic transformations due to their advantageous properties, such as non-flammability, negligible vapor pressure, high thermal and chemical stability, and ability to dissolve a wide range of materials (Chowdhury, Mohan, & Scott, 2007; Olivier-Bourbigou, Magna, & Morvan, 2010; Pârvulescu & Hardacre, 2007). ILs are miscible with materials having very wide range of polarities and are simultaneously able to dissolve a wide range of organic, inorganic and organometallic substances. These features offer numerous opportunities for the improvement of organic reactions using ILs as solvents and catalysts. Moreover, their ionic character enhances the reaction rates to a great extent in many reactions. Among them, Brønsted acidic ILs, especially the SO_3H-functionalized ones, have designed as environmentally friendly catalysts to replace the traditional mineral liquid acids like sulfuric acid and hydrochloric acid in chemical processes (Greaves & Drummond, 2008; Qiu et al., 2016; Shirole, Kadnor, Tambe, & Shelke, 2017; Vafaeezadeh & Alinezhad, 2016; Zolfigol, Khazaei, Moosavi-Zare, & Zare, 2010).

Considering the unique properties of Brønsted-acidic ILs, recently, we have synthesized two sulfonic acid functionalized ILs, including 1-methyl-1-sulfonic acid pyrrolidinium chloride [$MPyrrSO_3H$]Cl ($\mathbf{IL_1}$) and 4-methyl-4-sulfonic acid morpholinium chloride [$MMorSO_3H$]Cl ($\mathbf{IL_2}$) (Figure 1), and successfully applied them as highly efficient catalysts in the synthesis of 1,8-dioxooctahydroxanthenes (Dehghan, Davoodnia, Bozorgmehr, & Bamoharram, 2016). These findings encouraged us to explore other applications of these ILs in the synthesis of organic compounds. Therefore, in line with our interest on the development of convenient methods using reusable catalysts (Davoodnia, 2011; Davoodnia, Allameh, Fazli, & Tavakoli-Hoseini, 2011; Davoodnia, Basafa, & Tavakoli-Hoseini, 2016; Davoodnia, Khojastehnezhad, Bakavoli, & Tavakoli-Hoseini, 2011; Emrani, Davoodnia, & Tavakoli-Hoseini, 2011; Khashi, Davoodnia, & Prasada Rao Lingam, 2015; Moghaddas, Davoodnia, Heravi, & Tavakoli-Hoseini, 2012; Nakhaei & Davoodnia, 2014; Taghavi-Khorasani & Davoodnia, 2015), herein, we report the results of our investigation on the application of $\mathbf{IL_1}$ and $\mathbf{IL_2}$ as catalysts in the synthesis of amidoalkyl naphthols through the one-pot, three-component reaction of β-naphthol, aryl aldehydes, and acetamide under neat conditions (Scheme 1).

Figure 1. Structures of IL_1 and IL_2.

[MPyrrSO$_3$H]Cl (**IL$_1$**) [MMorSO$_3$H]Cl (**IL$_2$**)

Scheme 1. Synthesis of amidoalkyl naphthols catalyzed by Brønsted acidic ILs.



1 **2a-k** **3** **4a-k**

2. Results and discussion

As a preliminary, we directed our studies toward examination of the effect of various parameters like catalyst composition, effect of solvent, and influence of temperature on the reaction of β-naphthol (1) (1.0 mmol), 4-chlorobenzaldehyde (**2d**) (1.0 mmol), and acetamide (3) (1.0 mmol) for the synthesis of compound **4d** as the model reaction in the absence or presence of **IL$_1$** and **IL$_2$** as catalysts. A summary of the optimization experiments is provided in Table 1. First, to illustrate the need for catalyst in the reaction, the model reaction was studied in the absence of catalyst under solvent-free condition. The yield of the product was trace at 90°C after 60 min (Table 1, entry 1). Next, the reaction was performed in the presence of **IL$_1$** or **IL$_2$** in different solvents as well as under solvent-free conditions. Among the solvents tested, those being EtOH, MeOH, CH_2Cl_2, and MeCN, the reaction proceeded most readily to give the highest yield of the product **4d** under solvent-free conditions. It was observed that the yield of the final product **4d** increased with increasing amount of catalyst in the reaction mixture. The best result was obtained with 10 mol% of the catalyst under solvent-free conditions, which gave the desired product in 95 and 98% yields after 3 and 2 min at 90°C, respectively, for **IL$_1$** and **IL$_2$** (Table 1, entry 12). Further increase in temperature and **IL$_1$** or **IL$_2$** amount were found to have an inhibitory effect on formation of the product (Table 1, entries 13, 16, 17).

With the optimized conditions in hand, β-naphthol was reacted with acetamide and a wide variety of aromatic aldehydes using **IL$_1$** or **IL$_2$** (Table 2). As it can be seen, the reaction is effective with a variety of aromatic aldehydes with electron-donating or withdrawing substituents. Although the kind of aromatic aldehyde had no significant effect on the reaction, in most cases, but not all, aromatic aldehydes substituted with electron-withdrawing group or none reacted slightly faster than those with electron-donating groups and gave the higher yields of the products. Furthermore, both catalysts were highly efficient, and gave the desired amidoalkyl naphthols in high yields and short reaction times. However, as depicted, **IL$_2$** proved to be the better catalyst than **IL$_1$** in terms of yield and reaction time.

We also investigated recycling of the catalysts under solvent-free conditions using the model reaction. After completion of the reaction, the reaction mixture was cooled to room temperature, and warm distilled water was added. The product was collected by filtration, and washed repeatedly with warm distilled water. The combined filtrate was evaporated to dryness under reduced pressure. The residual ionic liquid was repeatedly washed with diethyl ether, dried under vacuum at 60°C, and used for the subsequent catalytic runs. The recovered catalyst worked well for up to five catalytic runs without any significant loss of its activity (95/98, 95/96, 93/95, 92/93, and 91/93% yields for **IL$_1$**/**IL$_2$** catalysts in first to fifth use, respectively).

Table 1. Screening of reaction condition for synthesis of compound 4d catalyzed by IL_1 or IL_2[a]

Entry	Catalyst (mol%)	Solvent	T (°C)	Time (min) IL_1/IL_2	Isolated yield (%) IL_1/IL_2
1	–	–	90	60/60	Trace/Trace
2	5	–	70	6/5	60/68
3	5	–	80	6/4	66/73
4	5	–	90	5/3	73/76
5	5	–	110	5/3	70/72
6	7	–	70	5/5	67/72
7	7	–	80	5/4	77/80
8	7	–	90	4/3	85/89
9	7	–	110	4/2	82/85
10	10	–	70	5/4	75/79
11	10	–	80	4/3	86/89
12	10	–	90	3/2	95/98
13	10	–	110	4/3	90/93
14	15	–	70	6/5	70/74
15	15	–	80	6/4	78/85
16	15	–	90	5/3	88/92
17	15	–	110	6/3	85/88
18	10	EtOH	Reflux	35/25	53/70
19	10	MeOH	Reflux	45/35	57/72
20	10	CH_2Cl_2	Reflux	40/30	54/60
21	10	MeCN	Reflux	30/20	65/72

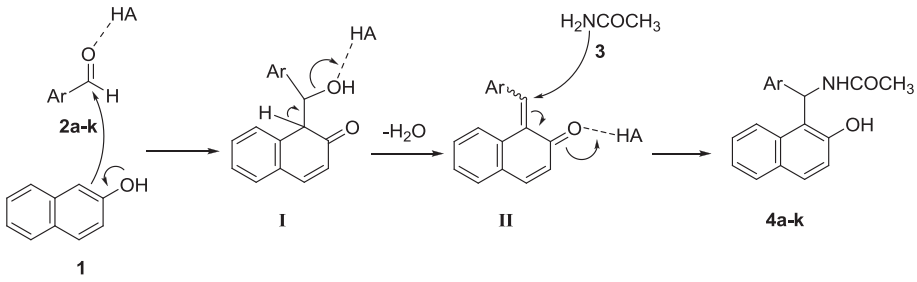
[a]Reaction conditions: β-naphthol (1) (1.0 mmol), 4-chlorobenzaldehyde (2d) (1.0 mmol), and acetamide (3) (1.0 mmol).

Table 2. IL_1 or IL_2 catalyzed synthesis of amidoalkyl naphthols (4a-k)[a]

Entry	Ar	Product	Time (min) IL_1/IL_2	Isolated yield (%) IL_1/IL_2	m.p. (°C)	lit. m.p. (°C)
1	C_6H_5	4a	4/2	94/97	242–244	240–242 (Kiasat et al., 2016)
2	4-$O_2NC_6H_4$	4b	4/2	89/91	246–248	242–244 (Kiasat et al., 2016)
3	3-$O_2NC_6H_4$	4c	6/3	86/90	239–241	241–243 (Kiasat et al., 2016)
4	4-ClC_6H_4	4d	3/2	95/98	228–230	225–227 (Kiasat et al., 2016)
5	2-ClC_6H_4	4e	4/3	86/91	206–208	204–205 (Shahrisa et al., 2012)
6	3-BrC_6H_4	4f	4/2	90/91	227–229	229–230 (Shahrisa et al., 2012)
7	4-BrC_6H_4	4g	5/2	92/94	227–228	230–232 (Davoodnia et al., 2014)
8	4-FC_6H_4	4h	4/3	93/95	224–226	226–228 (Davoodnia et al., 2014)
9	4-MeC_6H_4	4i	6/4	85/87	218–220	217–220 (Wang & Liang, 2011)
10	4-$MeOC_6H_4$	4j	5/3	80/83	182–184	180–182 (Wang & Liang, 2011)
11	3-Pyridyl	4k	6/5	86/89	190–192	192–194 (Bamoniri et al., 2014)

[a]Reaction conditions: β-naphthol (1) (1.0 mmol), an aromatic aldehyde (2a-k) (1.0 mmol), acetamide (3) (1.0 mmol), IL_1 or IL_2 (0.1 mmol, 10 mol%), 90°C, solvent-free.

Scheme 2. Plausible mechanism for the formation of amidoalkyl naphthols in the presence of IL_1 or IL_2 ≡ HA.



In accordance with the literature (Kiasat et al., 2016; Shahrisa et al., 2012), the suggested mechanism is described in Scheme 2. We believe that these ILs can act as Brønsted acids and therefore promotes the reactions by increasing the electrophilic character of the electrophiles in the reaction. At first, *ortho*-quinone methide (*o*-QM) intermediate [II] is readily formed *in situ* by Knoevenagel condensation of β-naphthol (1) and aromatic aldehydes (**2a-k**) via the intermediate [I]. Subsequent Michael addition of acetamide (3) to the *o*-QM intermediate [II] afforded the final products **4a-k**.

3. Conclusion

In conclusion, we showed that two newly synthesized Brønsted-acidic ILs, $\mathbf{IL_1}$ and $\mathbf{IL_2}$, efficiently catalyze the synthesis of amidoalkyl naphthols by increasing the electrophilic character of the electrophiles in the reaction β-naphthol, aryl aldehydes, and acetamide under solvent-free reactions. The kind of aldehyde had no significant effect on the reaction rates and products' yields. However, in general, electron-poor aldehydes reacted slightly faster than electron-rich ones and gave the higher yields of the products. Also, $\mathbf{IL_2}$ proved to be the better catalyst than $\mathbf{IL_1}$ in terms of yield and reaction time. Some advantages of this procedure are high yields, short reaction times, easy work-up, absence of volatile and hazardous solvents, and reusability of catalysts for a number of times without appreciable loss of activity.

4. Experimental

The $\mathbf{IL_1}$ and $\mathbf{IL_2}$ were synthesized according to the our previous report (Dehghan et al., 2016). All chemicals were available commercially and used without additional purification. Melting points were recorded on a Stuart SMP3 melting point apparatus. The ^{1}H NMR spectra were recorded with a Bruker 300 FT spectrometer.

4.1. General procedure for the synthesis of amidoalkyl naphthols (4a-k) catalyzed by $\mathbf{IL_1}$ or $\mathbf{IL_2}$

A mixture of β-naphthol (1) (1.0 mmol), an aromatic aldehyde (**2a-k**) (1.0 mmol), acetamide (3) (1.0 mmol), and $\mathbf{IL_1}$ or $\mathbf{IL_2}$ (0.1 mmol, 10 mol %) was heated in an oil bath at 90°C for 2–6 min. After completion of the reaction, monitored by TLC, the mixture was cooled to room temperature and warm distilled water was added. This resulted in the precipitation of the product, which was collected by filtration. The crude product was washed repeatedly with warm distilled water and then cold ethanol, and subsequently recrystallized from ethanol to give the pure products **4a-k** in high yields. The products were characterized according to comparison of their melting points with those of authentic samples and for some of them by their ^{1}H NMR spectral data.

4.2. Selected ^{1}H NMR data

N-((2-hydroxynaphthalen-1-yl)(phenyl)methyl)acetamide (**4a**): ^{1}H NMR (300 MHz, DMSO-d_6): δ 2.01 (s, 3H, CH_3), 7.10–7.45 (m, 9H, arom-H and CH_{Sp^3}), 7.76–7.87 (m, 3H, arom-H and NH), 8.52 (d, 1H, J = 8.1 Hz, arom-H), 10.08 (s br, 1H, OH).

N-((2-hydroxynaphthalen-1-yl)(4-nitrophenyl)methyl)acetamide (**4b**): ^{1}H NMR (300 MHz, DMSO-d_6): δ 2.04 (s, 3H, CH_3), 7.18–7.33 (m, 3H, arom-H and CH_{Sp^3}), 7.38–7.46 (m, 3H, arom-H), 7.78–7.88 (m, 3H, arom-H and NH), 8.16 (d, 2H, J = 9.0 Hz, arom-H), 8.62 (d, 1H, J = 7.8 Hz, arom-H), 10.01 (br, 1H, OH).

N-((4-Chlorophenyl)(2-hydroxynaphthalen-1-yl)methyl)acetamide (**4d**): ^{1}H NMR (300 MHz, DMSO-d_6): δ 2.03 (s, 3H, CH_3), 7.13–7.44 (m, 8H, arom-H and CH_{SP^3}), 7.82 (t, 2H, J = 8.7 Hz, arom-H), 7.87 (br, 1H, NH), 8.54 (d, 1H, J = 8.1 Hz, arom-H), 10.11 (s, 1H, OH).

N-((3-Bromophenyl)(2-hydroxynaphthalen-1-yl)methyl)acetamide (**4f**): ^{1}H NMR (300 MHz, DMSO-d_6): δ 2.00 (s, 3H, CH_3), 7.12 (d, 2H, J = 6.9 Hz, arom-H), 7.18–7.45 (m, 6H, arom-H and CH_{SP^3}), 7.77–7.90 (m, 3H, arom-H and NH), 8.51 (d, 1H, J = 8.4 Hz, arom-H), 9.69 (br, 1H, OH).

N-((4-Bromophenyl)(2-hydroxynaphthalen-1-yl)methyl)acetamide (**4g**): ^{1}H NMR (300 MHz, DMSO-d_6): δ 2.01 (s, 3H, CH_3), 7.08–7.32 (m, 5H, arom-H and CH_{SP^3}), 7.40 (t, 1H, J = 8.1 Hz, arom-H), 7.46 (d, 2H, J = 8.4 Hz, arom-H), 7.76–7.88 (m, 3H, arom-H and NH), 8.51 (d, 1H, J = 8.1 Hz, arom-H), 10.04 (s br, 1H, OH).

N-((2-hydroxynaphthalen-1-yl)(4-methoxyphenyl)methyl)acetamide (**4j**): ^{1}H NMR (300 MHz, DMSO-d_6): δ 2.00 (s, 3H, CH_3), 3.69 (s, 3H, OCH_3), 6.83 (d, 2H, J = 8.7 Hz, arom-H), 7.08–7.31 (m, 5H, arom-H and CH_{SP^3}), 7.38 (t, 1H, J = 7.2 Hz, arom-H), 7.75–7.94 (m, 3H, arom-H and NH), 8.49 (d, 1H, J = 8.4 Hz, arom-H), 10.05 (br, 1H, OH).

N-((2-hydroxynaphthalen-1-yl)(pyridin-3-yl)methyl)acetamide (**4k**): ^{1}H NMR (300 MHz, DMSO-d_6): δ 2.02 (s, 3H, CH_3), 7.15–7.35 (m, 4H, arom-H and CH_{SP^3}), 7.42 (t, 1H, J = 7.5 Hz, arom-H), 7.55 (d, 1H, J = 8.1 Hz, arom-H), 7.78–7.95 (m, 3H, arom-H and NH), 8.37–8.44 (m, 2H, arom-H), 8.57 (d, 1H, J = 8.1 Hz, arom-H), 10.14 (s br, 1H, OH).

Supplemental data
Supplemental data for this article can be accessed at http://dx.doi.org/10.1080/23312009.2017.1312675.

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