

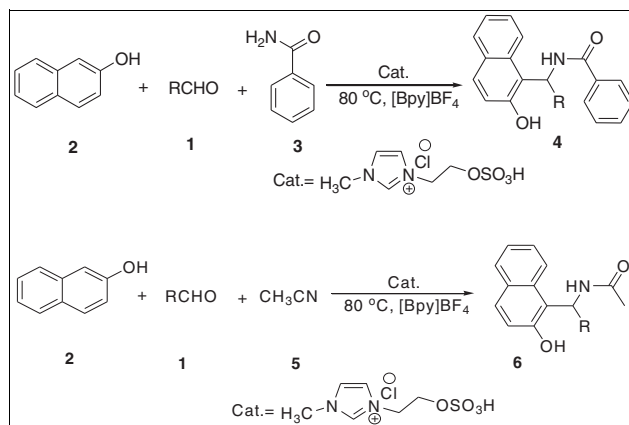
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The efficient one-pot syntheses of Betti bases by the three-component reaction of aromatic aldehyde, 2-naphthalen, and acetonitrile (or benzamide) catalyzed by 1-methyl-3-(2-(sulfooxy)ethyl)-1*H*-imidazol-3-ium chloride is reported. The solvent can be recycled easily.

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INTRODUCTION

The Betti bases (BT) chemistry has started at the beginning of the 20th century when Betti reported the synthesis of 1-(*a*-aminobenzyl)-2-naphthol [1]. The preparation of substituted Betti base derivatives by the modified Mannich reaction has subsequently become of considerable importance because a C–C bond is formed under mild experimental conditions [2]. In the past decade, interest in the Betti bases has turned to their application in chiral chemistry. The Betti bases and their *N*-substituted derivatives can serve as chiral catalysts [3–12], and the nonracemic Betti bases can be applied successfully as a new chiral auxiliary [13]. Chiral betti bases derivatives can also provide convenient access to many useful synthetic building blocks to afford a wide variety of chiral compounds [14].

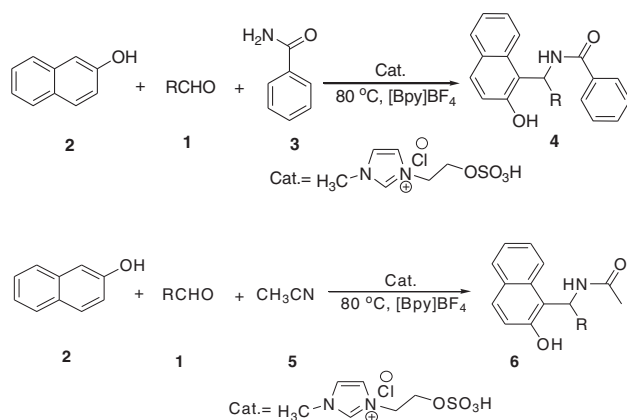
The base of obtaining chiral BT is the efficient synthesis of their racemic derivatives. The synthesis of racemic Betti bases has been generally promoted by acids, such as Fe(HSO₄)₃ [15], Ce(SO₄)₂ [16], CF₃COOH [17], I₂ [18,19], FeCl₃–SiO₂ [20], HClO₄–SiO₂ [21], NaHSO₄ [22], and ClSO₃H [23,24]. However, some shortcomings such as using violent toxic organic solvents and expensive, low-selective catalysts, rigorous condition, high reaction temperature, and long reaction times limited the scope of appropriate substrates. Therefore, introduction of clean

procedures and utilizing ecofriendly green catalyst can be simply recycled at the end of reaction have attracted more attention.

1-Methyl-3-(2-(sulfooxy)ethyl)-1*H*-imidazol-3-ium chloride (MSI) has been synthesized in our lab and proven as a very useful reagent in the preparation of some organic compounds, such as 14-aryl or alkyl-14*H*-dibenzo[*a,j*]xanthenes [25]. To research its new application, herein we described an MSI-catalyzed facile synthesis of Betti bases via a three-component reaction of aromatic aldehyde, 2-naphthalen, and acetonitrile (or benzamide) (Scheme 1).

RESULTS AND DISCUSSION

To optimize the reaction condition, the three-component reaction of *p*-nitrobenzaldehyde (**1h**), 2-naphthalen (**2**), and benzamide (**3**) was employed as a model reaction. The effects of catalysts were evaluated initially (Table 1). It was found that MSI was the most efficient catalyst (Table 1, Entries 1–7). The important role of MSI may be attributed to its fitting acidity, stability, and nonvolatility. Subsequently, the effects of solvents and reaction temperature were also evaluated. It was shown that when common organic solvents and water were used as solvent, the yields were very low (Table 1, Entries 8–12). It may be because

Scheme 1. One-pot synthesis of Betti bases catalyzed by MSI.

in these organic solvents, this reaction could only give some cross aldol condensation products. And these low-boiling organic solvents cannot offer enough energy to afford desired product that may be a thermodynamic-controlled one. Meanwhile, water may be making the equilibrium toward reactants. However, when three kinds of ionic liquids were used as reaction medium, the yield of **4h** increased to 83–95% surprisingly (Table 1, Entries 7, 13, and 14). [Bpy]BF₄ gave the highest yield because of its strongest basicity among these ionic

liquids. Finally, when the temperature was increased to 80 °C with [Bpy]BF₄ as the solvent, the reaction proceeded smoothly (Table 1, Entries 7 and 20–23). However, the increase in temperature did not enhance the yield of the product.

To explore the application of this method, the scope of the substrates was evaluated with a variety of aromatic or aliphatic aldehydes (Tables 2, 3). Obviously, the protocol was effective with aromatic aldehydes having either electron-withdrawing or electron-donating groups and also with aliphatic aldehydes. The electronic and steric effect of substituents in aromatic aldehydes had no significant effect on the yields. Also, we have investigated the reusability of [Bpy]BF₄ and observed that the ionic liquid was successfully reused for four cycles without significant loss of activity (Table 2, Entry 1).

Although the mechanism of this reaction remains to be fully clarified, the formation of compounds **4** and **6** could be explained by the sequence in Scheme 2. We supposed that the reaction may proceed via **A**, which was formed by the nucleophilic addition of 2-naphthol to aldehyde catalyzed by MSI. Subsequent Michael addition of **A** with the amide afforded the expected amidoalkyl naphthol (**4**). We discovered that when undehydrate acetonitrile was used as reagent, compound **6** can be obtained successfully. But when anhydrous acetonitrile was used, we can only obtain compound **7**. It is clear that in this process,

Table 1

Optimization of reaction conditions.

Entry	Catalyst	X (mol%)	Solvent	<i>t</i> (°C)	Yield (%)
1	I ₂	3	[Bpy]BF ₄	80	21
2	SSA	3	[Bpy]BF ₄	80	54
3	TsOH	3	[Bpy]BF ₄	80	80
4	HCl	3	[Bpy]BF ₄	80	11
5	SiO ₂	3	[Bpy]BF ₄	80	<5
6	HOAc	3	[Bpy]BF ₄	80	19
7	MSI	3	[Bpy]BF ₄	80	95
8	MSI	3	C ₂ H ₅ OH	80	51
9	MSI	3	THF	80	<5
10	MSI	3	DMF	80	43
11	MSI	3	H ₂ O	80	<5
12	MSI	3	Ethyl acetate	80	<5
13	MSI	3	[Bmim]BF ₄	80	83
14	MSI	3	[Bpy]Br	80	80
15	MSI	1	[Bpy]BF ₄	80	58
16	MSI	2	[Bpy]BF ₄	80	76
17	MSI	4	[Bpy]BF ₄	80	95
18	MSI	5	[Bpy]BF ₄	80	94
19	MSI	6	[Bpy]BF ₄	80	92
20	MSI	3	[Bpy]BF ₄	60	67
21	MSI	3	[Bpy]BF ₄	70	80
22	MSI	3	[Bpy]BF ₄	90	93
23	MSI	3	[Bpy]BF ₄	100	91

SSA, silica sulfuric acid; MSI, 1-methyl-3-(2-(sulfooxy)ethyl)-1*H*-imidazol-3-ium chloride.

All reactions were carried out in the scale of 2.0 mmol of *p*-nitrobenzaldehyde (**1h**), 2.0 mmol of 2-naphthalen (**2**), and 2.0 mmol of benzamide (**3**) in 25 min.

Table 2
Synthesis of **4** under optimum condition.

Entry	<i>R</i>	Time (min)	Yield (%)	mp (°C)	
				Found	Reported
4a	C ₆ H ₅	50	90 (90, 90, 89, 88) ^a	238–239	238–239 [27]
4b	3,4,5-(OCH ₃) ₃ C ₆ H ₂	60	85	229–230	235–238 [29]
4c	4-OCH ₃ C ₆ H ₄	60	89	211–212	208–209 [27]
4d	2,3-(CH ₃) ₂ C ₆ H ₃	30	87	223–224	–
4e	4-FC ₆ H ₄	50	90	233–234	233–234 [30]
4f	4-ClC ₆ H ₄	50	82	240–241	182–183 [29]
4g	2-OHC ₆ H ₄	40	84	250–251	–
4h	4-NO ₂ C ₆ H ₄	25	92	249–250	248–250 [28]
4i	2,3-(OCH ₃) ₂ C ₆ H ₃	35	90	225–226	–
4j	3-NO ₂ C ₆ H ₄	25	95	217–218	255–257 [29]
4k	2,4-ClC ₆ H ₃	40	86	235–236	263–264 [31]
4l	2-OCH ₃ C ₆ H ₄	40	82	220–221	266–267 [28]
4m	2-ClC ₆ H ₄	50	83	248–249	284–285 [31]
4n	2-BrC ₆ H ₄	50	85	239–240	228–230 [31]
4o	3-BrC ₆ H ₄	50	89	225–226	–
4p	3-FC ₆ H ₄	50	87	217–218	–
4q	3-ClC ₆ H ₄	50	83	236–237	–
4r	4-CHOC ₆ H ₄	60	91	257–258	–
4s	CH ₃ CH ₂	35	94	206–207	–

All reactions were carried out on a 2.0 mmol of aromatic aldehyde, 2.0 mmol of 2-naphthalen, and 2.0 mmol of benzamide.

^aYields after recovery of [Bpy]BF₄.

Table 3
Synthesis of **6** under optimum condition.

Entry	<i>R</i>	Time (h)	Yield (%)	mp (°C)	
				Found	Reported
6a	C ₆ H ₅	12	80	245–246	218–220 [28]
6b	4-CNC ₆ H ₄	10	86	251–252	232–234 [35]
6c	4-OCH ₃ C ₆ H ₄	12	81	183–184	183–184 [27]
6d	4-CH ₃ C ₆ H ₄	10	83	223–224	223–224 [27]
6e	4-FC ₆ H ₄	12	85	233–234	233–234 [31]
6f	4-ClC ₆ H ₄	12	84	223–224	230–231 [32]
6g	4-OHC ₆ H ₄	12	80	212–213	206–208 [37]
6h	4-IC ₆ H ₄	12	87	240–241	–
6i	2,3-(OCH ₃) ₂ C ₆ H ₃	12	82	236–237	235–236 [29]
6j	3-NO ₂ C ₆ H ₄	10	88	241–242	256–257 [27]
6k	2,4-ClC ₆ H ₃	12	82	201–202	201–203 [34]
6l	2-OCH ₃ C ₆ H ₄	12	81	208–209	241–242 [36]
6m	2-ClC ₆ H ₄	12	81	213–215	213–215 [32]
6n	2-BrC ₆ H ₄	12	83	220–221	228–230 [37]
6o	3-BrC ₆ H ₄	12	80	238–239	250–252 [37]
6p	3-FC ₆ H ₄	12	85	248–249	248–249 [34]
6q	4-BrC ₆ H ₄	12	83	227–229	228–230 [33]
6r	4-NO ₂ C ₆ H ₄	10	89	249–250	248–250 [34]

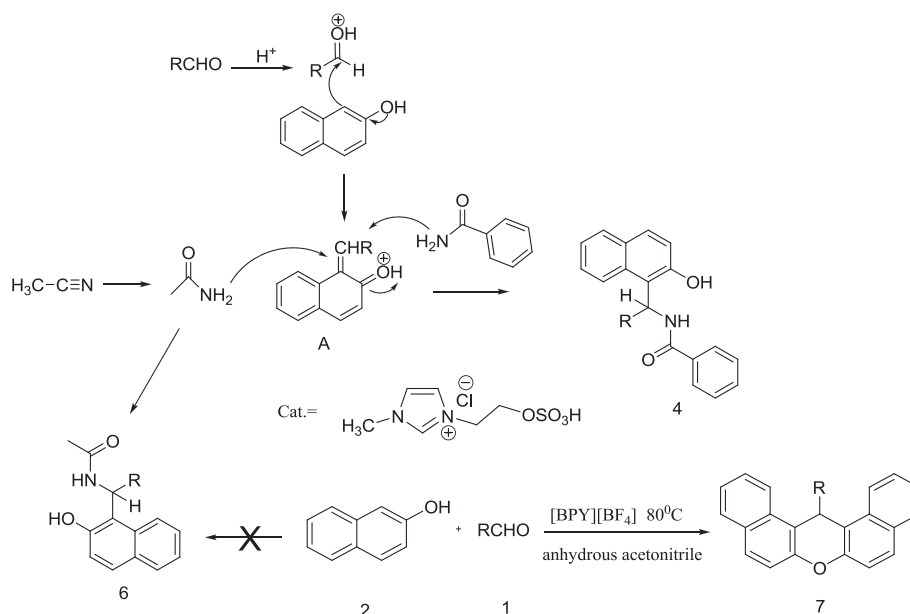
All reactions were carried out on a 2.0 mmol of aromatic aldehyde, 2.0 mmol of 2-naphthalen, and 2.0 mmol of acetonitrile.

acetonitrile undergoes hydrolysis to give acetamide, which further reacted with **A** to give **6**.

In summary, we have described the synthesis of Betti bases via the one-pot three-component reaction of aldehyde, 2-naphthalen, and acetonitrile (or benzamide) catalyzed by MSI. This new simple method paves the way for the synthesis of chrial Betti bases.

EXPERIMENTAL

General information. Commercial solvents and reagents were used as received. Melting points were uncorrected. IR spectra were recorded on Varian F-1000 spectrometer in KBr with absorptions in cm⁻¹. ¹H NMR was determined on Varian-400 MHz spectrometer in DMSO-*d*₆. (*J* values are in Hz). Chemical shifts were expressed in parts per million downfield from

Scheme 2. Proposed mechanism for the synthesis of 1-methyl-3-(2-(sulfooxy)ethyl)-1*H*-imidazol-3-ium chloride.

internal standard TMS. The synthesis of MSI was followed by the literated method [26].

The procedure for the synthesis of 4. A mixture of aldehyde (2 mmol), 2-naphthalen (2 mmol), benzamide (2 mmol), ionic liquid [Bpy]BF₄ (2 mL), and MSI (0.06 mmol) was stirred at 80°C for 25–60 min (monitored by TLC). The resulting solid was washed with water and dried in vacuum. The crude product was purified by recrystallization from ethanol and DMF to give 4.

The procedure for the synthesis of 6. A mixture of aldehyde (2 mmol), 2-naphthalen (2 mmol), acetonitrile (2 mmol), ionic liquid [Bpy]BF₄ (2 mL), and MSI (0.06 mmol) was stirred at 80°C for 10–12 h (monitored by TLC). The resulting solid was washed with water and dried in vacuum. The crude product was purified by recrystallization from ethanol and DMF give to 6.

The spectral data of new products are the following.

***N*-((2-hydroxynaphthalen-1-yl)(2,3-dimethylphenyl)methyl) benzamide (4d).** mp 223–224°C, white solid; IR (KBr, ν , cm⁻¹): 3423, 3158, 3027, 1630, 1573, 1539, 1488, 1344, 1271, 1075, 942, 819, 754; ¹H NMR (400 MHz, DMSO-*d*₆) δ 10.04 (s, 1H), 8.94 (d, *J* = 8.0 Hz, 1H), 8.00 (d, *J* = 8.8 Hz, 1H), 7.86 (d, *J* = 7.2 Hz, 2H), 7.75–7.81 (m, 2H), 7.50 (t, *J* = 7.2 Hz, 1H), 7.36–7.44 (m, 3H), 7.32 (d, *J* = 7.2 Hz, 1H), 7.20–7.28 (m, 2H), 7.07 (d, *J* = 8.0 Hz, 1H), 6.90–6.96 (m, 2H), 2.26 (d, *J* = 4.8 Hz, 6H); HRMS (ESI) *m/z*: calcd for [M+Na]⁺ C₂₆H₂₃NO₂: 404.1770; found: 404.1774.

***N*-((2-hydroxynaphthalen-1-yl)(2-hydroxyphenyl)methyl) benzamide (4g).** mp 250–251°C, white solid; IR (KBr, ν , cm⁻¹): 3426, 3140, 3065, 1634, 1573, 1540, 1488, 1272, 1056, 823, 753, 711; ¹H NMR (400 MHz, DMSO-*d*₆) δ 9.99 (s, 1H), 9.01 (d, *J* = 7.6 Hz, 1H), 8.07 (d, *J* = 8.8 Hz, 1H), 7.88 (d, *J* = 7.6 Hz, 2H), 7.76–7.82 (m, 2H), 7.51 (t, *J* = 7.2 Hz, 1H), 7.41–7.49 (m, 6H), 7.34 (d, *J* = 7.6 Hz, 1H), 7.24–7.29 (m, 3H), 7.17 (d, *J* = 8.8 Hz, 1H); HRMS (ESI) *m/z*: calcd for [M+Na]⁺ C₂₄H₁₉NO₃: 393.3100; found: 393.3104.

***N*-((2-hydroxynaphthalen-1-yl)(2,3-dimethoxyphenyl)methyl) benzamide (4i).** mp 225–226°C, white solid; IR (KBr, ν , cm⁻¹): 3424, 3129, 3058, 1633, 1573, 1537, 1479, 1263, 1065, 1001, 820, 755, 709; ¹H NMR (400 MHz, DMSO-*d*₆) δ 10.20 (s, 1H), 8.94 (d, *J* = 8.8 Hz, 1H), 8.20 (d, *J* = 8.8 Hz, 1H), 7.85 (d, *J* = 8.4 Hz, 2H), 7.73–7.80 (m, 2H), 7.43–7.52 (m, 5H), 7.28 (t, *J* = 7.9 Hz, 1H), 7.19 (d, *J* = 8.8 Hz, 1H), 7.10 (d, *J* = 6.8 Hz, 1H), 6.91–6.98 (m, 2H), 3.77 (s, 3H), 3.55 (s, 3H); HRMS (ESI) *m/z*: calcd for [M+Na]⁺ C₂₆H₂₃NO₄: 436.1696; found: 436.1696.

***N*-((3-bromophenyl)(2-hydroxynaphthalen-1-yl)methyl) benzamide (4o).** mp 225–226°C, white solid; IR (KBr, ν , cm⁻¹): 3394, 3197, 3065, 1627, 1576, 1508, 1474, 1269, 1053, 805, 823, 692, 634; ¹H NMR (400 MHz, DMSO-*d*₆) δ 10.38 (s, 1H), 9.05 (d, *J* = 8.0 Hz, 1H), 8.08 (d, *J* = 8.8 Hz, 1H), 7.81–7.88 (m, 4H), 7.56 (t, *J* = 7.6 Hz, 1H), 7.48 (t, *J* = 8.0 Hz, 3H), 7.41–7.44 (m, 2H), 7.29–7.34 (m, 2H), 7.23–7.26 (m, 3H); HRMS (ESI) *m/z*: calcd for [M+Na]⁺ C₂₄H₁₈BrNO₂: 454.0560; found: 454.0585.

***N*-((3-fluorophenyl)(2-hydroxynaphthalen-1-yl)methyl) benzamide (4p).** mp 217–218°C, white solid; IR (KBr, ν , cm⁻¹): 3414, 3177, 3071, 1627, 1573, 1538, 1480, 1261, 1127, 936, 815, 740, 689; ¹H NMR (400 MHz, DMSO-*d*₆) δ 10.37 (s, 1H), 9.04 (d, *J* = 8.0 Hz, 1H), 8.09 (d, *J* = 8.8 Hz, 1H), 7.81–7.89 (m, 4H), 7.56 (t, *J* = 7.6 Hz, 1H), 7.49 (t, *J* = 7.6 Hz, 3H), 7.29–7.35 (m, 3H), 7.25 (d, *J* = 8.8 Hz, 1H), 7.02–7.11 (m, 3H); HRMS (ESI) *m/z*: calcd for [M+Na]⁺ C₂₄H₁₈FNO₂: 394.1398; found: 394.1381.

***N*-((3-chlorophenyl)(2-hydroxynaphthalen-1-yl)methyl) benzamide (4q).** mp 236–237°C, white solid; IR (KBr, ν , cm⁻¹): 3397, 3198, 3067, 1627, 1576, 1508, 1475, 1270, 1166, 1054, 806, 729, 681; ¹H NMR (400 MHz, DMSO-*d*₆) δ 10.37 (s, 1H), 9.04 (d, *J* = 8.4 Hz, 1H), 8.08 (d, *J* = 8.4 Hz, 1H), 7.81–7.88 (m, 4H), 7.56 (t, *J* = 7.6 Hz, 1H), 7.46–7.51 (m, 3H), 7.29–7.34 (m, 5H), 7.22–7.27 (m, 2H); HRMS (ESI) *m/z*: calcd for [M+Na]⁺ C₂₄H₁₈ClNO₂: 410.1081; found: 410.1089.

N-((4-formylphenyl)(2-hydroxynaphthalen-1-yl)methyl)benzamide (**4r**). mp 257–258°C, white solid; IR (KBr, ν , cm^{-1}): 3419, 3196, 3071, 1633, 1576, 1516, 1436, 1268, 1054, 809, 732, 623; ^1H NMR (400 MHz, $\text{DMSO-}d_6$) δ 10.37 (s, 1H), 9.00 (d, $J=4.4$ Hz, 1H), 8.07 (d, $J=8.0$ Hz, 1H), 7.76–7.82 (m, 5H), 7.53 (t, $J=7.2$ Hz, 1H), 7.44 (t, $J=8.0$ Hz, 4H), 7.29 (d, $J=7.6$ Hz, 1H), 7.19–7.24 (m, 5H); HRMS (ESI) m/z : calcd for $[\text{M}+\text{Na}]^+$ $\text{C}_{25}\text{H}_{19}\text{NO}_3$: 404.1771; found: 404.1766.

N-(1-(2-hydroxynaphthalen-1-yl)propyl)benzamide (**4s**). mp 206–207°C, white solid; IR (KBr, ν , cm^{-1}): 3405, 3178, 3068, 1635, 1574, 1514, 1436, 1348, 1174, 1073, 816, 748, 707; ^1H NMR (400 MHz, $\text{DMSO-}d_6$) δ 10.09 (s, 1H), 8.61 (d, $J=8.4$ Hz, 1H), 8.22 (d, $J=8.8$ Hz, 1H), 7.78–7.81 (m, 3H), 7.70 (d, $J=8.8$ Hz, 1H), 7.44–7.52 (m, 5H), 7.29 (t, $J=7.6$ Hz, 1H), 7.18 (d, $J=8.8$ Hz, 1H), 2.11–2.17 (m, 1H), 1.92–2.02 (m, 1H), 0.92 (t, $J=7.6$ Hz, 3H); HRMS (ESI) m/z : calcd for $[\text{M}+\text{Na}]^+$ $\text{C}_{20}\text{H}_{19}\text{NO}_2$: 328.1477; found: 328.1478.

N-((4-cyanophenyl)(2-hydroxynaphthalen-1-yl)methyl)acetamide (**6b**). mp 251–252°C, white solid; IR (KBr, ν , cm^{-1}): 3379, 3108, 2958, 2231, 1636, 1576, 1509, 1438, 1280, 1063, 819, 752, 594; ^1H NMR (400 MHz, $\text{DMSO-}d_6$) δ 10.20 (s, 1H), 8.56 (d, $J=8.0$ Hz, 1H), 7.80–7.84 (m, 3H), 7.73 (d, $J=8.0$ Hz, 2H), 7.40 (t, $J=6.4$ Hz, 1H), 7.27–7.34 (m, 3H), 7.22 (d, $J=9.2$ Hz, 1H), 7.15 (d, $J=8.0$ Hz, 1H), 2.01 (s, 3H); IR (KBr, ν , cm^{-1}): 3379, 3108, 2958, 2231, 1636, 1576, 1509, 1438, 1280, 1063, 819, 752, 594; HRMS (ESI) m/z : calcd for $[\text{M}+\text{H}]^+$ $\text{C}_{20}\text{H}_{16}\text{N}_2\text{O}_2$: 318.3080; found: 318.3096.

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