Chemistry of Heterocyclic Compounds, Vol. 47, No. 9, December, 2011 (Russian Original Vol. 47, No. 9, September, 2011)

A CONVENIENT METHOD FOR THE SYNTHESIS OF 1-R-1,2,3,4-TETRAHYDROBENZOTHIENO-[2,3-*c*]PYRIDINE DERIVATIVES

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New 1-R-2-methyl-1,2,3,4-tetrahydrobenzothieno[2,3-*c*]pyridine derivatives have been obtained by cyclization of 2-(1-benzothiophen-3-yl)ethylamines with aromatic and heteroaromatic aldehydes in the presence of triisopropylchlorosilane.

Keywords: 2-(1-benzothiophen-3-yl)ethylamine, 1,2,3,4-tetrahydrobenzothieno[2,3-*c*]pyridine, triisopropylchlorosilane, Pictet-Spengler reaction.

Benzothieno[2,3-*c*]pyridines, which are *S*-isosteres of β-carbolines, possess high biological activity. Testing of benzothiophene analogs harmine and harmaline as MAO inhibitors *in vitro* showed that the effects of harmine and *S*-harmine are nearly identical, while *S*-harmaline is 50 times as active as harmaline. Sulfur analogs of *Harmala* alkaloids have higher lipid solubility, shorter biological half-lives, and less bond to tissue than their nitrogen-containing analogs [1].

Among benzothieno[2,3-*c*]pyridines, analgesics, tranquilizers, antidepressants, α_2-blockers, and anorexic compounds were found. Analysis of the patent literature shows that for most benzothieno[2,3-*c*]pyridine derivatives psychotropic effects are expressed characteristically [2].

One of the most widespread synthetic approach to 1,2,3,4-tetrahydrobenzothieno[2,3-*c*]pyridines is based on closure of the pyridine ring using the Pictet-Spengler reaction [2-5]. However the cyclization of 2-(1-benzothiophen-3-yl)ethylamines derivatives with formaldehyde in concentrated hydrochloric acid yielding 1,2-unsubstituted 1,2,3,4-tetrahydrobenzothieno[2,3-*c*]pyridines is described in most papers. Two examples of obtaining their 1-methyl- and 1-phenyl derivatives are described in papers [3, 4]. Besides, a two-step method for the preparation of 1-phenyl derivatives is recommended involving the preliminary preparation of azomethines followed by cyclization in hydrochloric acid. *N*-Substituted 1,2,3,4-tetrahydrobenzothieno[2,3-*c*]pyridines were obtained by alkylation or acylation of NH group [5-7]. There are no examples in the literature for the use of *N*-substituted 2-(1-benzothiophen-3-yl)ethylamines in the Pictet-Spengler reaction. Our interest toward *N*-substituted benzothienopyridines is also connected with the preparation of benzothienoazocines on their basis [8-10].

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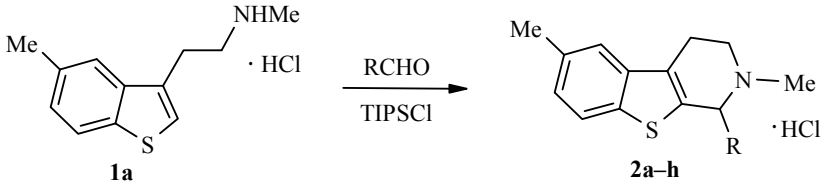
Translated from Khimiya Geterotsiklicheskikh Soedinenii, No. 9, 1418-1424, September, 2011. Original article submitted December 28, 2010. Revision submitted September 6, 2011.

A previously described method for the synthesis of benzothieno[2,3-*c*]pyridines [3, 4] gives low yields of final cyclization products as for *N*-methyl-2-(5-methyl-1-benzothiophen-3-yl)ethylamine **(1a)**.

Trimethylchlorosilane–dimethyl formamide was recently proposed as a new reagent for acid-catalyzed process (such as the Biginelli and Friedlander reactions) [11, 12]. We successfully applied this methodology to prepare 2-methyl-1,2,3,4-tetrahydrobenzothieno[2,3-*c*]pyridines **2a-b** by the Pictet-Spengler reaction from amine **1a** and aromatic or heterocyclic aldehydes. Triisopropylchlorosilane (TIPSCl) was used instead of trimethylchlorosilane that allowed to expand the temperature range for the reaction. The starting hydrochloride **1a** was dissolved in DMF, and the quantity used as a rule did not exceed 1.5-2 ml per 2 mmol of the starting amines **1a,b.**



1a **2a–h**

2 a R = Ph, **b** R = 4-MeOC$_6$H$_4$, **c** R = 4-ClC$_6$H$_4$, **d** R = 3-FC$_6$H$_4$, **e** R = 3-Py, **f** R = 2-thienyl, **g** R = 3-bromo-2-thienyl, **h** R = 1-benzofuran-2-yl

TABLE 1. Characteristics of Compounds **2a-b, 3a-c, 4a-c**

Compound	Empirical formula	Found, % / Calculated, %				mp, °C, (mp, °C, of hydrochlorides)	Yield, %
		C	H	N	S		
2a	$C_{19}H_{19}NS$	77.89 / 77.77	6.65 / 6.53	4.70 / 4.77	10.81 / 10.93	124-125 (241-242)	83
2b	$C_{20}H_{21}NOS$	74.38 / 74.27	6.62 / 6.54	4.21 / 4.33	9.80 / 9.91	125-126 (225-226)	71
2c	$C_{19}H_{18}ClNS$	69.52 / 69.60	5.60 / 5.53	4.15 / 4.27	9.69 / 9.78	152-153 (254-255)	75
2d	$C_{19}H_{18}FNS$	73.39 / 73.28	5.90 / 5.83	4.65 / 4.50	10.22 / 10.30	104-105 (238-239)	83
2e	$C_{18}H_{18}N_2S$	73.58 / 73.43	6.08 / 6.16	9.60 / 9.51	10.73 / 10.89	105-106 (240-241)	88
2f	$C_{17}H_{17}NS_2$	68.11 / 68.19	5.66 / 5.72	4.73 / 4.68	21.49 / 21.41	124-125 (151-152)	72
2g	$C_{17}H_{16}BrNS_2$	53.88 / 53.97	4.20 / 4.26	3.61 / 3.70	17.02 / 16.95	154-155 (186-187)	92
2h	$C_{21}H_{19}NOS$	75.73 / 75.64	5.69 / 5.74	4.11 / 4.20	9.54 / 9.62	149-150 (215-216)	78
3a	$C_{17}H_{16}ClNS$	67.54 / 67.65	5.27 / 5.34	4.57 / 4.64	10.71 / 10.62	(283-284)	62
3b	$C_{18}H_{18}ClNOS$	65.04 / 65.15	5.42 / 5.47	4.14 / 4.22	9.75 / 9.66	(255-256)	67
3c	$C_{17}H_{15}ClN_2O_2S$	58.73 / 58.87	4.41 / 4.36	8.15 / 8.08	9.18 / 9.24	(234-235)	68
4a	$C_{18}H_{18}ClNS$	68.58 / 68.45	5.80 / 5.74	4.38 / 4.43	10.09 / 10.15	(214-215)	84
4b	$C_{19}H_{20}ClNOS$	65.85 / 65.98	5.78 / 5.83	4.11 / 4.05	9.18 / 9.27	(205-206)	68
4c	$C_{18}H_{17}ClN_2O_2S$	60.04 / 59.91	4.70 / 4.75	7.82 / 7.76	8.81 / 8.89	(191-192)	87

TABLE 2. ^{1}H NMR Spectra of Compounds **2a-b, 3a-c, 4a-c**

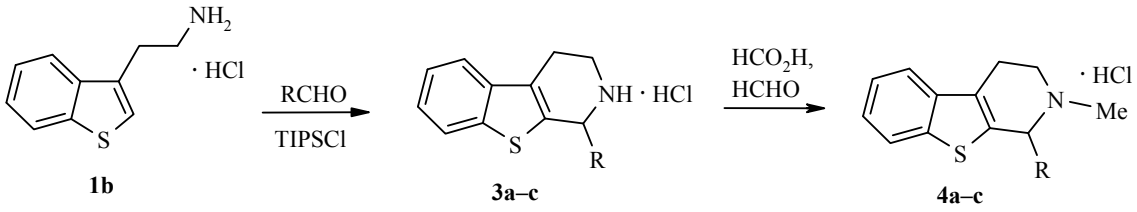
Compound	Chemical shifts, δ, ppm (*J*, Hz)
2a	2.34 (3H, s, 6-CH_3); 2.46 (3H, s, 2-CH_3); 2.77, 2.88, 3.09, and 3.26 (4H, four m, CH_2CH_2); 4.38 (1H, s, 1-CH); 7.08 (1H, d, *J* = 8.0, H-7); 7.29-7.35 (5H, m, H Ph); 7.43 (1H, s, H-5); 7.54 (1H, d, *J* = 8.0, H-8)
2b	2.32 (3H, s, 6-CH_3); 2.47 (3H, s, 2-CH_3); 2.76, 2.87, 3.06, and 3.25 (4H, four m, CH_2CH_2); 3.81 (3H, s, 4'-OCH_3); 4.33 (1H, s, 1-CH); 6.87 (2H, d, *J* = 8.0, H-3',5'); 7.08 (1H, d, *J* = 8.0, H-7); 7.27 (2H, d, *J* = 8.0, H-2',6'); 7.42 (1H, s, H-5); 7.55 (1H, d, *J* = 8.0, H-8)
2c	2.29 (3H, s, 6-CH_3); 2.44 (3H, s, 2-CH_3); 2.74, 2.86, 3.04, and 3.22 (4H, four m, CH_2CH_2); 4.34 (1H, s, 1-CH); 7.08 (1H, d, *J* = 8.0, H-7); 7.29 (4H, br. s, H-2',3',5',6'); 7.41 (1H, s, H-5); 7.53 (1H, d, *J* = 8.0, H-8)
2d	2.36 (3H, s, 6-CH_3); 2.49 (3H, s, 2-CH_3); 2.79, 2.90, 3.08, and 3.27 (4H, four m, CH_2CH_2); 4.42 (1H, s, 1-CH); 7.02 (1H, t, *J* = 8.0, H-5'); 7.12 (2H, m, H-7,4'); 7.19 (1H, d, *J* = 8.0, H-6'); 7.33 (1H, m, H-2'); 7.45 (1H, s, H-5); 7.57 (1H, d, *J* = 8.0, H-8)
2e	2.35 (3H, s, 6-CH_3); 2.48 (3H, s, 2-CH_3); 2.81, 2.91, 3.10, and 3.26 (4H, four m, CH_2CH_2); 4.46 (1H, s, 1-CH); 7.12 (1H, d, *J* = 8.0, H-7); 7.27 (1H, t, *J* = 4.8, H-5'); 7.45 (1H, s, H-5); 7.57 (1H, d, *J* = 8.0, H-8); 7.68 (1H, d, *J* = 4.8, H-4'); 8.58 (1H, d, *J* = 4.8, H-6'); 8.62 (1H, s, H-2')
2f	2.46 (3H, s, 6-CH_3); 2.47 (3H, s, 2-CH_3); 2.82, 2.89, 3.02, and 3.30 (4H, four m, CH_2CH_2); 4.86 (1H, s, 1-CH); 6.98 (1H, t, *J* = 4.0, H-4'); 7.12 (2H, m, H-5',7); 7.30 (1H, d, *J* = 4.0, H-3'); 7.43 (1H, s, H-5); 7.59 (1H, d, *J* = 8.0, H-8)
2g	2.47 (3H, s, 6-CH_3); 2.48 (3H, s, 2-CH_3); 2.83, 2.90, 2.97, and 3.26 (4H, four m, CH_2CH_2); 4.81 (1H, s, 1-CH); 7.03 (1H, s, H-5'); 7.14 (1H, d, *J* = 8.0, H-7); 7.20 (1H, s, H-4'); 7.43 (1H, s, H-5); 7.61 (1H, d, *J* = 8.0, H-8)
2h	2.49 (3H, s, 6-CH_3); 2.55 (3H, s, 2-CH_3); 2.90, 2.98, 3.02, and 3.32 (4H, four m, CH_2CH_2); 4.94 (1H, s, 1-CH); 6.70 (1H, s, H-3'); 7.14 (1H, d, *J* = 8.0, H-7); 7.22-7.27 (2H, m, H-5',6'); 7.46 (2H, m, H-5,4'); 7.54 (1H, d, *J* = 8.0, H-8); 7.61 (1H, d, *J* = 8.0, H-7')
3a	3.17, 3.36, 3.47, 3.61 (4H, four m, CH_2CH_2); 5.90 (1H, s, 1-CH); 7.36 (1H, t, *J* = 8.0, H-7); 7.43 (1H, t, *J* = 8.0, H-6); 7.45 (3H, m, H-3',4',5'); 7.61 (2H, m, H-2',6'); 7.76 (1H, d, *J* = 8.0, H-8); 7.79 (1H, d, *J* = 8.0, H-5); 10.65 (1H, br. s, NH)
3b	3.15, 3.36, 3.44 and 3.59 (4H, four m, CH_2CH_2); 3.84 (3H, s, OCH_3); 5.83 (1H, s, 1-CH); 6.95 (2H, d, *J* = 8.0, H-3',5'); 7.35 (1H, t, *J* = 8.0, H-7); 7.42 (1H, t, *J* = 8.0, H-6); 7.51 (2H, d, *J* = 8.0, H-2',6'); 7.74 (1H, d, *J* = 8.0, H-8); 7.78 (1H, d, *J* = 8.0, H-5); 10.16 (1H, br. s, NH)
3c	3.19, 3.40, 3.52 and 3.65 (4H, four m, CH_2CH_2); 6.19 (1H, s, 1-CH); 7.38 (1H, t, *J* = 8.0, H-7); 7.45 (1H, t, *J* = 8.0, H-6); 7.78 (1H, d, *J* = 8.0, H-8); 7.81 (1H, d, *J* = 8.0, H-5); 7.95 (2H, d, *J* = 8.0, H-2',6'); 8.30 (2H, d, *J* = 8.0, H-3',5'); 10.63 (1H, br. s, NH)
4a	2.77 (3H, s, 2-CH_3); 3.18-3.86 (4H, m, CH_2CH_2); 5.92 (1H, s, 1-CH); 7.36 (1H, t, *J* = 8.0, H-7); 7.44 (1H, t, *J* = 8.0, H-6); 7.50 (3H, m, H-3',4',5'); 7.73 (2H, m, H-2',6'); 7.76 (1H, d, *J* = 8.0, H-8); 7.79 (1H, d, *J* = 8.0, H-5)
4b	2.77 (3H, s, 2-CH_3); 3.14-3.70 (4H, m, CH_2CH_2); 3.85 (3H, s, OCH_3); 5.82 (1H, br. s, 1-CH); 6.98 (2H, d, *J* = 8.0, H-3',5'); 7.35 (1H, t, *J* = 8.0, H-7); 7.42 (1H, t, *J* = 8.0, H-6); 7.65 (2H, d, *J* = 8.0, H-2',6'); 7.75 (2H, d, *J* = 8.0, H-5,8)
4c	2.78 (3H, s, 2-CH_3); 3.20, 3.52, 3.67, and 3.87 (4H, four m, CH_2CH_2); 6.20 (1H, s, 1-CH); 7.37 (1H, t, *J* = 8.0, H-7); 7.44 (1H, t, *J* = 8.0, H-6); 7.78 (2H, d, *J* = 8.0, H-5,8); 8.07 (2H, d, *J* = 8.0, H-2',6'); 8.30 (2H, d, *J* = 8.0, H-3',5')

At a temperature of 130-140°C, the reaction was completed in 2 h. In some cases, for example to obtain compounds **2a-c**, increasing the temperature to 160°C shortened the reaction time to 30 min. After cooling, the crystalline mass was well washed with dry acetone and ether. As a rule, the 2-methyl-1,2,3,4-tetrahydrobenzothieno[2,3-*c*]pyridine hydrochlorides did not require additional purification and were spectroscopically

pure. The method is limited to aromatic and heterocyclic aldehydes. Aliphatic aldehydes, such as propanal and butanal, did not give tetrahydrobenzothienopyridine derivatives and only the starting hydrochloride **1a** was isolated. 2-(5-Chloro-1-benzothiophen-3-yl)-1-methylethylamine did not gave 1,2,3,4-tetrahydrobenzothieno-[2,3-*c*]pyridines even after prolonged heating of the reaction mixture at 150°C due to electron-withdrawing influence of the chlorine atom in position 5 of the benzothiophene ring.

The advantage of TIPSCl as a catalyst in the Pictet-Spengler reaction is possibility to use heteroaromatic aldehydes, for example, thiophene-2-aldehyde, which rapidly polymerizes in concentrated hydrochloric acid.

This method was also used for the synthesis of 2-unsubstituted 1,2,3,4-tetrahydrobenzothieno[2,3-*c*]-pyridines **3a-c**. In this case 2-(1-benzothiophen-3-yl)ethylamine (**1b**) was used as the starting material. Methylation of 1,2,3,4-tetrahydrobenzothieno[2,3-*c*]pyridines **3a-c** was carried out by the Eschweiler-Clarke reaction with a mixture of 90% formic acid and formaldehyde.



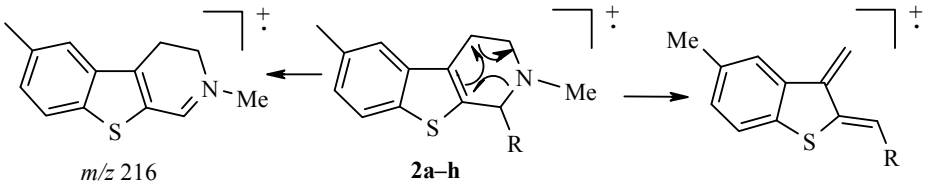
1b **3a–c** **4a–c**

3, 4 a R = Ph, **b** R = 4-$MeOC_6H_4$, **c** R = 4-$NO_2C_6H_4$

In the ^{1}H NMR spectra (Table 2) of 1,2,3,4-tetrahydrobenzothieno[2,3-*c*]pyridines **2-4** each of the protons in CH_2CH_2N fragment is observed as a free standing multiplet.

As can be seen from the data presented in Table 3, the basic factor influencing the stability of the molecules of the synthesized compounds to the electron impact is the presence of a substituent on the nitrogen atom. Mass spectra of the 2-unsubstituted 1,2,3,4-tetrahydrobenzothieno[2,3-*c*]pyridines are characterized by molecular ion peaks with maximum intensity, whereas introduction of a methyl substituent in position 2 leads to an increased extent of fragmentation. This peculiarity is probably explained by the ease of the primary fragmentation, connected with rupture of the C(1)–N(2) bond with subsequent rupture of the tetrahydropyridine ring with elimination of the $HN{=}CH_2$ fragment (compounds **3a-c**) or the $MeN{=}CH_2$ unit (compounds **4a-c**). Evidently, this rupture would occur more easily at a tertiary nitrogen as a result of participation of the substituent in stabilization of the cation formed. Elimination of these fragments probably occurs by a mechanism analogous to the retrodiene reaction which is a characteristic of 1,2,3,4-tetrahydrodibenzothiophenes [14, 15].

The basic direction of the fragmentation of the *N*-substituted pyridines **2a-h** $[M]^+$ ions is connected with elimination of the fragment R and formation of an ion with *m/z* 216, which peak has the maximum intensity in the mass spectra of compounds **2c-e.** As for compounds **2f-h** the dominating primary route of fragmentation becomes the opening of the tetrahydropyridine ring *via* a retrodiene mechanism:



m/z 216 **2a–h**

Thus we described a convenient route to produce 1-R-1,2,3,4-tetrahydrobenzothieno[2,3-*c*]pyridines in high yields *via* the Pictet-Spengler reaction in the presence of triisopropylchlorosilane as a catalyst.

TABLE 3. Mass Spectra of Compounds **2a-b, 3a-c, 4a-c**

Compound	*m/z* (I_{rel}, %)*
2a*	294 $[M+1]^+$ (100)
2b*	324 $[M+1]^+$ (100)
2c	329 $[M]^+$ (19), 328 (21), 327 $[M]^+$ (49), 326 (29), 285 (13), 284 (23), 250 (10), 249 (58), 248 (15), 234 (20), 217 (16), 216 (100), 152 (24), 124 (12), 42 (33)
2d	312 (12), 311 $[M]^+$ (63), 310 (34), 269 (11), 268 (52), 267 (26), 253 (10), 252 (16), 217 (13), 216 (100), 136 (20), 42 (19)
2e	294 $[M]^+$ (50), 251 (48), 250 (57), 217 (25), 216 (100), 119 (22), 42 (23)
2f	299 $[M]^+$ (91), 257 (19), 256 (100), 255 (40), 241 (12), 240 (16), 216 (40), 211 (12), 124 (33), 42 (20)
2g	379 $[M]^+$ (77), 378 (45), 377 $[M]^+$ (76), 376 (33), 337 (12), 336 (71), 335 (13), 334 (68), 256 (20), 255 (100), 254 (19), 240 (25), 222 (41), 217 (13), 216 (93), 204 (25), 202 (28), 149 (22), 127 (17), 108 (10), 42 (43)
2h	333 $[M]^+$ (40), 332 (19), 291 (21), 290 (100), 216 (15), 158 (11)
3a	266 (34), 265 $[M]^+$ (100), 264 (80), 236 (18), 235 (18), 234 (14), 188 (52), 162 (14), 104 (11)
3b	296 (21), 295 $[M]^+$ (100), 294 (84), 266 (11), 235 (11), 188 (25), 162 (12), 134 (14)
3c	311 (21), 310 $[M]^+$ (100), 309 (55), 264 (12), 263 (19), 235 (20), 234 (31), 188 (46), 162 (12), 147 (14), 117 (10)
4a	279 $[M]^+$ (53), 278 (33), 236 (48), 235 (29), 234 (13), 203 (15), 202 (100), 118 (20), 42 (30)
4b	310 (22), 309 $[M]^+$ (97), 308 (62), 267 (10), 266 (46), 265 (24), 235 (42), 234 (11), 221 (12), 203 (14), 202 (100), 155 (11), 148 (35), 42 (29)
4c	324 $[M]^+$ (51), 322 (23), 281 (38), 264 (17), 235 (23), 234 (37), 203 (13), 202 (100), 163 (19), 42 (34)

* Mass spectra were obtained by the APCI method.

EXPERIMENTAL

^{1}H NMR spectra were recorded on a Bruker DRX 400 (400 MHz) instrument in $CDCl_3$ (compounds **2a-h**) and in DMSO-d_6 (compounds **3a-c, 4a-c** as hydrochlorides) with TMS as internal standard. EI mass spectra were recorded on an M1321 mass spectrometer with an ionization voltage of 70 eV (ionization chamber at 220°C) with direct insertion of the sample. Chromato-mass spectra were recorded on an Agilent 1100 LC/MSD VL spectrometer with APCI ionization (chemical positive ionization at atmospheric pressure). Parameters of the chromatographic column: length 50 mm, diameter 4.6 mm, stationary phase ZORBAX SB-C18, solvent acetonitrile–water 95:5, 0.1% trifluoroacetic acid, gradient elution, rate of flow of solvent 3.0 ml/min. Characteristics of the synthesized compounds are given in Tables 1-3.

***N*-Methyl-2-(5-methyl-1-benzothiophen-3-yl)ethylamine (1a)** and **2-(1-benzothiophen-3-yl)ethylamine (1b)** were obtained by reduction of the corresponding amides of 1-benzothiophene-3-acetic acid with diborane in THF [16].

1,2,3,4-Tetrahydrobenzothieno[2,3-*c*]pyridines 2a-h, 3a-c (General Method). A mixture of DMF (2 ml), corresponding hydrochloride **1a,b** (2 mmol), aldehyde (2.1 mmol), and TIPSCl (10 mmol) was heated for 2 h in a standard 8 ml ampule at 140°C. The ampule was cooled and the precipitate triturated with dry

acetone (5 ml) and dry ether (3 ml). The precipitate was filtered off and washed with warm acetone. The bases were obtained by basifying aqueous–ethanolic solutions of the hydrochlorides with 5% NaOH solution. The bases were crystallized from 50% aqueous ethanol.

2-Methyl-1,2,3,4-tetrahydrobenzothieno[2,3-*c*]pyridines 4a-c (General Method). A mixture of hydrochloride **3a-c** (2 mmol), 90% formic acid (4 ml), 37% formaldehyde (4 ml), and water (4 ml) was boiled for 2 h. The mixture was cooled and added to 60 ml 1 M NaOH solution. The precipitate was filtered off, washed with water, dried, and converted to the hydrochloride with concentrated HCl in ethanol. The products were crystallized from ethanol.

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