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Facile Synthesis of Azaspirocycles via Iron Trichloride-Promoted Cyclization/ Chlorination of Cyclic 8-Aryl-5-aza-5-tosyl-2-en-7-yn-1-ols

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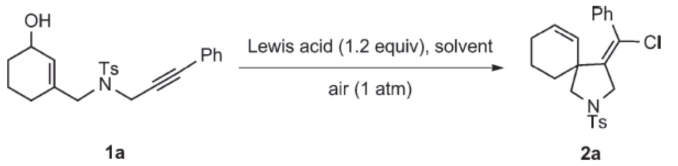
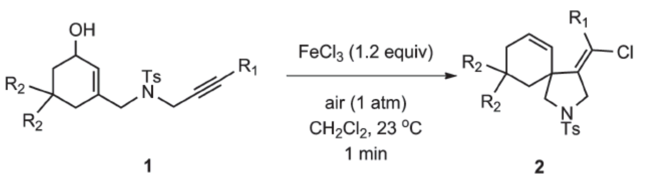
Table 1. Optimizing the Reaction Conditions



entry	Lewis acid	solvent	temp (°C)	time	yield (%)
1	$FeCl_3$	CH_2Cl_2	23	1 min	83
2	$FeCl_3$	CH_2Cl_2	0	3 min	72
3	$AlCl_3$	CH_2Cl_2	23	1 min	67[a]
4	$TiCl_4$	CH_2Cl_2	23	0.5 h	17[b]
5	$SnCl_4$	CH_2Cl_2	23	0.5 h	27
6	$ZnCl_2$	CH_2Cl_2	23	48 h	0
7	$Fe(NO_3)_3 \cdot 9H_2O$	CH_2Cl_2	23	48 h	0
8	$FeCl_3$	DCE	23	1 min	74
9	$FeCl_3$	DBE	23	2 h	72
10	$FeCl_3$	THF	23	26 h	23
11	$FeCl_3$	CH_3CN	23		0

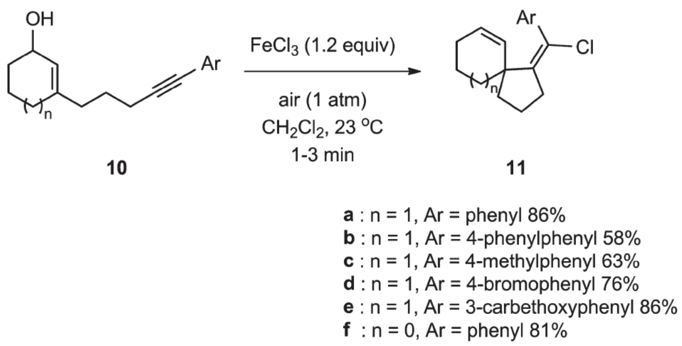
[a] $Z/E = 6{:}1$ (determined by 400 MHz ^{1}H NMR analysis of the crude reaction mixture). [b] $Z/E = 13{:}1$ (determined by 400 MHz ^{1}H NMR analysis of the crude reaction mixture).

Table 2. $FeCl_3$-Promoted Cyclization/Chlorination of Various Cyclic 8-Aryl-5-aza-5-tosyl-2-en-7-yn-1-ols



entry	substrate	R_1	R_2	product	yield (%)
1	**1a**	phenyl	H	**2a**	83
2	**1b**	4-methylphenyl	H	**2b**	80
3	**1c**	4-nitrophenyl	H	**2c**	83
4	**1d**	3-carbethoxyphenyl	H	**2d**	89
5	**1e**	4-phenylphenyl	H	**2e**	89
6	**1f**	4-bromophenyl	H	**2f**	90
7	**1g**	phenyl	CH_3	**2g**	97
8	**1h**	4-methoxyphenyl	H		
9	**1i**	H	H		

Scheme 3. $FeCl_3$-Promoted Cylization/Chlorination of **10**



Scheme 1. Plausible Mechanism