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Ytterbium (III) triflate catalysed [3 + 2] cycloaddition involving isothiocyanates and epichlorohydrin

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Highly regioselective cycloadditions occur in reactions of substituted isothiocyanates and epichlorohydrin to give *N*-(5-(chloromethyl)-1,3-oxathiolan-2-ylidene)anilines catalysed by $Yb(OTf)_3$. The configuration is retained at the chiral centres of the epoxides.

Keywords: isothiocyanate, epichlorohydrin, ytterbium triflate

Epoxides are useful intermediates in organic synthesis because of their easy accessibility and high reactivity. A number of syntheses of five-membered heterocycles from epoxides have been reported in the literature.[1-4] Baba *et al.*[4] have reported the cycloaddition of epoxides with heterocumulenes promoted by high-coordinated trialkyltin complexes, whilst Shibata *et al.*[3] have reported the same reactions using organotin iodine-Lewis base complexes as catalysts. We have reported the cycloaddition of epichlorohydrin with thioureas[5] or N-arylimines[6] catalysed by lanthanide triflates. Compared with the other catalysts, rare earth metal triflates have been found to be unique Lewis acids in that they are water tolerant, recyclable catalysts that can effectively promote several carbon–carbon and carbon–heteroatom bond formation reactions.[7-9] During our ongoing research on the application of epoxides in organic synthesis,[5,6] we have been interested in finding a simple cycloaddition of isothiocyanates with epichlorohydrin **1a** using lanthanide triflates as catalysts.

Our primary experiments were performed using isothiocyanate **2a** as a model reaction (Scheme 1). Initially, the [3 + 2] cycloaddition reaction was carried out in the presence of $Yb(OTf)_3$ under solvent-free conditions at room temperature for 72 hours. Unfortunately, the desired product was obtained with only 26% yield. So we optimised the solvents to increase the yields (Table 1).

It was found that solvents play an important role in the cycloaddition reaction. When the reaction is carried out in DMF, the desired product was obtained in moderate yield, but THF, CH_2Cl_2, toluene, 1,4-dioxane, CH_3NO_2 and H_2O resulted in no desired products. Ionic liquids, such as $[BMIM][PF_6]$ and [BPy]Br, were also used but no desired product was detected.

Several Lewis acids were examined to promote this cycloaddition (Table 2). It was found that $Yb(OTf)_3$ could promote this reaction efficiently, while $BiCl_3$, $Zn(OTf)_2$ $Sr(OTf)_2$ and $AlCl_3$ were less effective. When $Cu(OTf)_2$ was used, no products were detected. Higher regioselectivity products were formed in the presence of $Yb(OTf)_3$ (**3a**:**4a** = 90:10, Table 2 entry 4). However, other catalysts only provided products with poor regioselectivity. Then the amounts of catalyst were investigated. It was found that 5 mol% was enough to promote this cycloaddition and no evident improvement of yields was observed by increasing the amount of catalyst up to 20 mol%. So we chose 5 mol% $Yb(OTf)_3$ to catalyse this reaction.

Table 1 Effect of solvents in [3 + 2] cycloaddition of **1a** with **2a**[a]

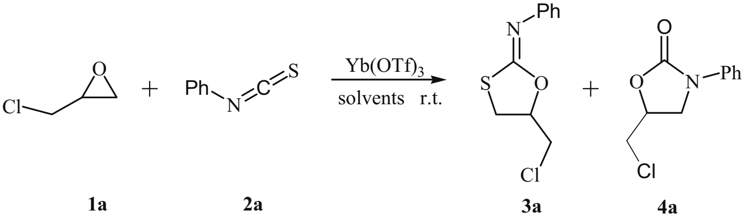
Entry	Solvent	Time/h[b]	Yield/%[c]
1	THF	72	0
2	Toluene	72	0
3	1,4-Dioxane	72	0
4	CH_3CH_2OH	72	0
5	CH_2Cl_2	72	0
6	CH_3NO_2	72	0
7	H_2O	72	0
8	$[BMIM][PF_6]$	72	0
9	[BPy]Br	72	0
10	Solvent-free	72	26 (88 : 12)
11	DMF	44	52 (90 : 10)

[a] Reaction conditions: 10 mol% $Yb(OTf)_3$, r.t.
[b] The reaction was monitored by TLC.
[c] Isolated yields based on **2a**.

Table 2 Effect of catalyst in [3 + 2] cycloaddition of **1a** with **2a**[a]

Entry	Catalyst	Loading/mol%	Time/h[b]	Yield/%[c]
1	None		72	27 (60:40)
2	$Yb(OTf)_3$	1	75	35 (90:10)
3	$Yb(OTf)_3$	5	14	73 (88:12)
4	$Yb(OTf)_3$	10	12	72 (90:10)
5	$Yb(OTf)_3$	15	12	69 (91:9)
6	$Yb(OTf)_3$	20	12	70 (90:10)
7	Sr(OTf)	10	35	21 (70:30)
8	$Sc(OTf)_3$	10	15	65 (85:15)
9	$Zn(OTf)_2$	10	35	41 (70:30)
10	$Cu(OTf)_2$	10	10	ND[d]
11	$BiCl_3$	10	70	32 (63:37)
12	$AlCl_3$	10	65	29 (57:43)

[a] Reaction conditions: 60 °C in DMF.
[b] The reaction was monitored by TLC.
[c] Isolated yields based on **2a**.
[d] No desired product was obtained.



Scheme 1

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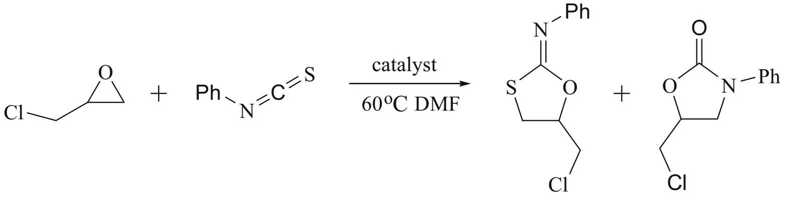


1a **2a** **3a** **4a**

Scheme 2

On the other hand, the reaction temperature obviously affected the results. Optimal isolated yield (up to 73%) on **2a** was obtained when the reaction mixture was stirred for 14 h at 60 °C. In the procedure of our experiment, we found that when the temperature was higher than 80 °C, much more side-reactions took place with some by-products. Excess epichlorohydrin **1a** was proved to be necessary because it was easy to polymerise in the presence of Lewis acid catalysts.

To explore the scope of our method, a variety of isothiocyanates were investigated to react with epichlorohydrin catalysed by Yb(OTf)$_3$ in DMF. The structure of products **3** and **4** were fully identified by ^{1}H NMR, ^{13}C NMR, MS and IR spectra. The results are in Table 3.

No desired products were detected when R^2 are alkyl groups (Table 3, Entries 14 and 15) due to the lower stability of the alkyl substituted products. Changing the substituent groups on the isothiocyanates showed significant effects on the reaction. Generally, electron-withdrawing groups gave poor yields (Table 3, Entries 9 and 10), while electron-donating groups gave good yields (Table 3, Entry 2). The position of substituent groups was also important. For example, the *p*-Me substituted isothiocyanates gave the better yields than *m*-Me and *o*-Me. (Table 3, Entries 2, 3 and 6).

In the case of 4-chlorophenyl-isothiocyanates (Table 3, entry 9), not only product **3i** was detected, but also an unexpected by-product was obtained (Scheme 4) under similar conditions. Thiazolidinimines could be synthesised from epoxycholopropane and N-aryl substituted thiourea, which was obtained through hydrolysis of isothiocyanates, catalysed by Yb(OTf)$_3$ in DMF[5].

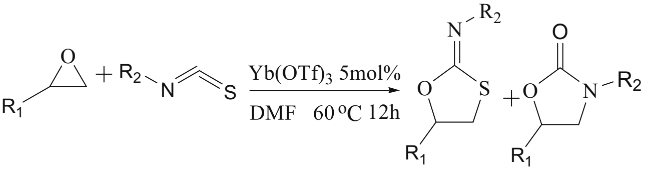
To our surprise, when the reaction proceeded with 2,5-diisopropylphenyl-isothiocyanate and epoxycholopropane under the above conditions, the main product was **6** instead of **3h** (Scheme 5). This phenomenon was not observed in any other reaction even when reaction time was prolonged. It may be ascribed to the steric effect.

Reaction of chiral (R)-epichlorohydrin **1a** and phenyl isothiocyanates **2a** afforded (R)-products under the same conditions (Scheme 6). It was found that the absolute configuration at the more substituted C-α position of the epoxide was retained during the reaction. An optically active product with 99%ee was obtained, which was checked by chiral HPLC analysis. HPLC condition: DAICEL CHIRALCELOD-H-4.6 × 250 mm, hexane: C_2H_5OH = 85: 15(% V/V), 254nm, 0.5 mL/min, 30 °C.

Table 3 Cyclisation of oxiranes with isothiocyanates[a,b]

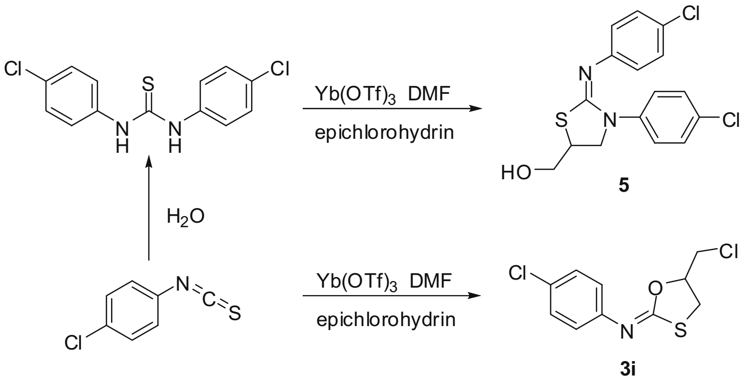
Entry	R^1	R$^{2\,c}$	Product	Time/h	Yield **3**(%)[d]
1	CH_2Cl	C_6H_5	**3a, 4a**	12	73 (88:12)
2	CH_2Cl	*p*-Me-C_6H_4	**3b**	12	67 (100:0)
3	CH_2Cl	*m*-Me-C_6H_4	**3c**	15	62 (100:0)
4	CH_2Cl	*p*-CH_3O-C_6H_4	**3d**	12	63 (100:0)
5	CH_2Cl	*2,4*-$Me_2C_6H_3$	**3e**	12	57 (100:0)
6	CH_2Cl	*o*-Me-C_6H_4	**3f**	18	55 (100:0)
7	CH_2Cl	*o*-ethyl-C_6H_4	**3g**	17	50 (100:0)
8	CH_2Cl	*2,6*-(*i*-Pr)$_2C_6H_3$	**6**	45	48 [g]
9	CH_2Cl	*p*-Cl-C_6H_4	**3i, 5**	5	35 (72:28)[g]
10	CH_2Cl	*p*-F-C_6H_4	**3j**	17	32 (100:0)
11	(R)-CH_2Cl	C_6H_5	**3k, 4k**	12	70 (90:10)
12	Ph	C_6H_5	None		0
13	$C_6H_5OCH_2CH_2$	C_6H_5	None		0
14	CH_2Cl	Cyclohexyl	None		0
15	CH_2Cl	*n*-Bu	None		0

[a] Reaction conditions: 1 (10 mmol), 2 (2 mmol), DMF (5 mL), The reactions were carried out at 60 °C.
[b] The reaction was monitored by TLC.
[c] Isothiocyanates were prepared according to the literature.[12]
[d] Isolated yields based on 2.
[g] Different product was obtained.

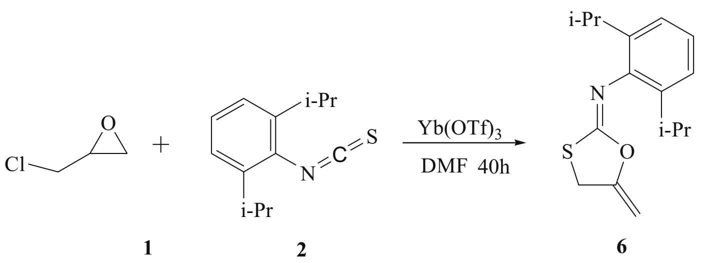


1 **2** **3** **4**

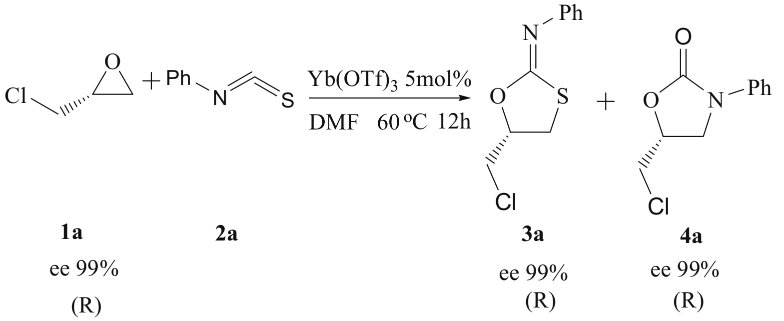
Scheme 3



Scheme 4



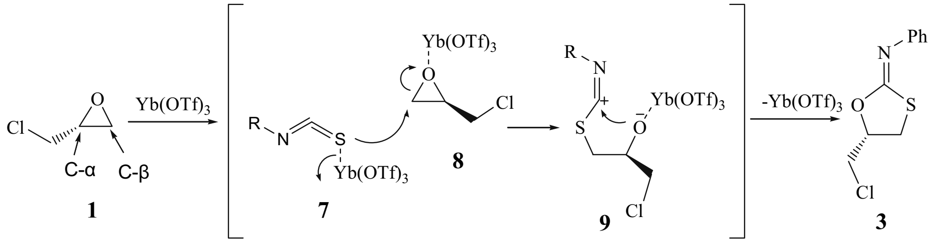
Scheme 5



Scheme 6

With the above results in hand, the possible mechanism of this cyclisation reaction was postulated in Scheme 7. The epoxide **1** was coordinated to $Yb(OTf)_3$ to generate an intermediate **8**. Then **8** was attacked by carbon–sulfur double bond of the isothiocyanate moiety. These reactions gave a highly reactive intermediate **9**, which then undergoes a [3 + 2] cycloaddition reaction to provide the substituted 2-oxathiolanimines **3**. The nucleophilic attack happened at C-ß of the epoxide (nonsubstituted side), and the absolute configuration at the more substituted C-α position of the epoxide was retained during the transformations. Also the ring opening occurred at the C-ß position in this reaction. So we obtained products with complete regioselectivity.

In summary, we have demonstrated that $Yb(OTf)_3$ is an effective catalyst for the [3 + 2] cycloaddition of epichlorohydrin and isothiocyanates with high regioselectivity. It



Scheme 7

provides a novel synthetic method for the construction of five-membered heterocycles.

Experimental

The NMR spectra were measured with a Bruker Advance III 500 or Varian Mercury Plus-400 instrument using CDCl$_3$ as the solvent with TMS as internal standard. IR spectra were recorded using KBr pellets on a Nicolet Aviatar-370 instrument. Mass spectra were measured with Thermo Finnigan LCQ-Advantage. High resolution Mass spectra were measured on Bruker APEX III spectrometer using EI techniques.

General procedure for preparation of **3a–k**

Phenyl isothiocyanate **2a** (2 mmol) was added to a stirred mixture of DMF (5 mL) and epoxide **1a** (10 mmol). The reaction was monitored by TLC until completed. The preparation of **3a** was kept at 60 °C and maintained for 12 h. Then the solvent was removed by distillation at reduced pressure. The residue was purified on silica gel (petroleum ether:EtOAc:CH$_2$Cl$_2$ 10: 1: 1) to give **3a** and **4a**. The physical and spectra data of the compounds **3a–k** are as follows:

N-(5-(Chloromethyl)-1,3-oxathiolan-2-ylidene)aniline **(3a):** Wax. IR (cm^{-1}): 1655 (C=N). ^{1}H NMR δ: 7.32 (t, 2H, J = 7.6 Hz), 7.12 (t, 1H, J = 7.6 Hz), 6.96 (d, 1H, J = 8.4 Hz), 4.81–4.87 (m,1H), 3.74–3.84 (m, 2H), 3.50 (dd, 1H, J_1 = 6.4, J_2 = 11.2 Hz), 3.40 (dd, 1H, J_1 = 7.2, J_2 = 11.2 Hz). ^{13}C NMR: δ 33.8, 42.8, 79.8, 121.1 × 2, 124.5, 129.1 × 2, 148.5, 162.3. m/z (EI): 227, 229(M$^+$). HRMS-EI: Calcd for C$_{10}$H$_{10}$NOSCl: 227.0172. Found: 227.0173.

5-(Chloromethyl)-3-phenyloxazolidin-2-one **(4a):** Solid. M.p. 99.2–100.3 °C.[3] IR (cm^{-1}): 1738 (C=O). ^{1}H NMR δ: 7.55 (d, 2H, J = 4.4 Hz), 7.38–7.41 (m, 2H), 7.17 (t, 1H, J = 7.2 Hz), 4.87 (t, 1H, J = 2.8 Hz), 4.16–4.19 (m, 1H), 3.97 (dd, 1H, J_1 = 5.6, J_2 = 9.2 Hz), 3.73–3.83 (m, 2H). ^{13}C NMR δ: 44.5, 48.1, 70.9, 118.3 × 2, 124.3, 129.1 × 2, 137.8, 153.9. m/z (EI): 211, 213(M$^+$). HRMS-EI: Calcd for C$_{10}$H$_{10}$NO$_2$Cl: 211.0400. Found: 211.0406.

N-(5-(Chloromethyl)-1,3-oxathiolan-2-ylidene)-4-methyl-benzenamine **(3b):** Wax. IR (cm^{-1}): 1667 (C=N). ^{1}H NMR δ: 7.15 (d, 2H, J = 7.5 Hz), 6.89 (d, 2H, J = 7.5 Hz), 4.85–4.90 (m, 1H), 3.77–3.87 (m, 2H), 3.54 (dd, 1H, J_1 = 6.0, J_2 = 11.0 Hz), 3.43 (dd, 1H, J_1 = 7.0, J_2 = 11.0 Hz), 2.35(s, 3H). ^{13}C NMR δ: 22.7, 29.7, 42.7, 79.7, 120.9 × 2, 129.8 × 2, 134.1, 146.0, 162.2. m/z (EI): 241, 243(M$^+$). HRMS-EI: Calcd for C$_{11}$H$_{12}$NOSCl: 241.0328. Found: 241.0334.

N-(5-(Chloromethyl)-1,3-oxathiolan-2-ylidene)-3-methylbenzenamine **(3c)***:* Wax. IR (cm^{-1}): 1668 (C=N). ^{1}H NMR δ: 7.21 (t, 1H, J = 6.0 Hz), 6.94 (d, 1H, J = 6.0 Hz), 4.82–4.86 (m, 1H), 3.82 (dd, 1H, J_1 = 4.5, J_2 = 10.5 Hz), 3.76 (dd, 1H, J_1 = 7.5, J_2 = 11.5 Hz), 3.49 (dd, 1H, J_1 = 6.0, J_2 = 11.0 Hz), 3.39 (dd, 1H, J_1 = 7.0, J_2 = 11.0 Hz). ^{13}C NMR δ 21.4, 33.9, 42.8, 79.8, 118.0, 121.8, 125.3, 129.0, 139.1, 148.6, 162.2. m/z (EI): 241, 243(M$^+$). HRMS-EI: Calcd for C$_{11}$H$_{12}$NOSCl: 241.0328. Found: 241.0334.

N-(5-(Chloromethyl)-1,3-oxathiolan-2-ylidene)-4-methoxy-benzenamine **(3d)**: Wax. IR (cm^{-1}): 1665 (C=N). ^{1}H NMR δ: 6.90–6.92(m,2H), 6.85–6.88(m, 2H), 4.82–4.87 (m,1H), 3.83 (dd, 1H, J_1=4.5, J_2=11.5 Hz), 3.78 (s, 3H), 3.76 (dd, 1H, J_1=7.5, J_2=11.5 Hz), 3.52 (dd, 1H, J_1 = 6.5, J_2 = 11.5 Hz), 3.41 (dd, 1H, J_1 = 7.0, J_2 = 11.5 Hz). ^{13}C NMR δ: 33.9, 43.0, 55.5, 79.7, 114.0, 114.4, 122.2 × 2, 141.8, 156.6 162.2. m/z (EI): 257, 259(M$^+$). HRMS-EI: Calcd for C$_{11}$H$_{12}$NO$_2$SCl: 257.0277. Found: 257.0281.

N-(5-(Chloromethyl)-1,3-oxathiolan-2-ylidene)-2,4-dimethylaniline **(3e):** Wax. IR (cm^{-1}): 1672 (C=N). ^{1}H NMR δ: 7.00 (s, 1H), 6.96 (d, 1H, J = 6.0 Hz), 6.74 (d, 1H, J = 8.0), 4.84–4.90 (m, 1H), 3.85 (dd, 1H, J_1 = 4.5, J_2 = 11.5 Hz), 3.77 (dd, 1H, J_1 = 8.0, J_2 = 11.0 Hz), 3.52 (dd, 1H, J_1 = 6.5, J_2 = 11.5 Hz), 3.41(dd, 1H, J_1 = 7.0, J_2 = 11.0 Hz), 2.30(s, 3H), 2.16(s, 3H). ^{13}C NMR δ: 17.7, 20.9, 33.9, 42.8, 79.9, 119.8, 127.1, 129.6, 131.3, 134.0, 145.0, 162.30. m/z (EI): 255(M$^+$). HRMS-EI: Calcd for C$_{12}$H$_{14}$NOSCl: 255.0485. Found: 255.0491.

N-(5-(Chloromethyl)-1,3-oxathiolan-2-ylidene)-2-methylaniline **(3f):** Wax. IR (cm^{-1}): 1660 (C=N). ^{1}H NMR δ: 7.14–7.19 (m, 2H), 7.04 (t, 1H, J = 7.5 Hz), 6.84 (d, 1H, J = 4.0 Hz), 4.86–4.89 (m, 1H), 3.849 (dd, 1H, J_1 = 4.5, J_2 = 11.5 Hz), 3.79 (dd, 1H, J_1 = 7.5, J_2 = 11.0 Hz), 3.52 (dd, 1H, J_1 = 6.5, J_2 = 11.5 Hz), 3.41 (dd, 1H, J_1 = 7.0, J_2 = 11.0 Hz), 2.19 (s, 3H) ^{13}C NMR δ: 17.7, 33.8, 42.8, 79.9, 120.0, 124.5, 126.6, 130.5, 143.9, 163.8. m/z (EI): 241, 243(M$^+$). HRMS-EI: Calcd for C$_{11}$H$_{12}$NOSCl: 241.0328. Found: 241.0334.

N-(5-(Chloromethyl)-1,3-oxathiolan-2-ylidene)-2-ethylaniline **(3g):** Wax. IR (cm^{-1}): 1667 (C=N). ^{1}H NMR δ: 7.06–7.25 (m, 3H), 6.83 (d, 1H, J = 7.6), 4.83 (m, 1H), 3.84 (dd, 1H, J_1 = 4.4, J_2 = 11.2 Hz), 3.77 (dd, 1H, J_1 = 7.6, J_2 = 11.2 Hz), 3.51 (dd, 1H, J_1 = 6.0, J_2 = 10.8 Hz), 3.95 (dd, 1H, J_1 = 7.6, J_2 = 15.2 Hz), 2.54–2.60 (m, 2H), 1.16 (t, 3H, J = 7.2). ^{13}C NMR δ: 14.6, 24.5, 33.8, 42.8, 79.9, 120.2, 124.7, 126.5, 128.7, 135.9, 147.0, 161.8. m/z (EI): 255, 257(M$^+$). HRMS-EI: Calcd for C$_{12}$H$_{14}$NOSCl: 255.0485. Found: 255.0484.

2,6-Disopropyl-N-(5-methylene-1,3-oxathiolan-2-ylidene) benzenamine **(6):** Solid M.p. 121.1–122.8 °C. IR (cm^{-1}): 1663 (C=N). ^{1}H NMR δ: 7.33 (t, 1H, J = 8.0 Hz), 7.21 (d, 2H, J = 6.4 Hz), 6.49–6.50 (m, 1H), 6.01–6.05 (m, 1H), 4.17 (dd, 2H, J_1 = 1.5, J_2 = 4.5 Hz), 2.96–3.01 (m, 2H), 1.22 (dd, 12H, J_1 = 6.5, J_2 = 8.5 Hz). ^{13}C NMR δ: 24.2 × 4, 28.8 × 2, 52.7, 118.2, 123.2, 124.5 × 2, 129.0, 137.6, 145.9, 162.9. m/z (EI): 275(M$^+$). HRMS-EI: Calcd for C$_{16}$H$_{21}$NOS: 275.1344. Found: 275.1343.

4-Chloro-N-(5-(chloromethyl)-1,3-oxathiolan-2-ylidene) benzenamine **(3i):** Wax. IR (cm^{-1}): 1666 (C=N). ^{1}H NMR δ: 7.26–7.30 (m, 2H), 6.88–6.91 (m, 2H), 4.84–4.92 (m, 1H), 3.83 (dd, 1H, J_1 = 4.5, J_2 = 11.5 Hz), 3.77 (dd, 1H, J_1 = 7.5, J_2 = 11.5 Hz), 3.54 (dd, 1H, J_1 = 6.5, J_2 = 11.5 Hz), 3.43 (dd, 1H, J_1 = 7.0, J_2 = 11.0 Hz). ^{13}C NMR δ: 34.0, 42.7, 80.0, 122.6, 124.4, 128.7, 129.3, 129.8, 147.1, 163.0. m/z (EI): 261, 263, 265(M$^+$). HRMS-EI: Calcd for C$_{10}$H$_9$NOSCl$_2$: 260.9782. Found: 260.9785.

N-(5-(Chloromethyl)-1,3-oxathiolan-2-ylidene)-4-fluoro-benzenamine **(3j):** Wax. IR (cm^{-1}): 1665 (C=N). ^{1}H NMR δ: 7.01–7.04 (m, 2H), 6.92–6.94 (m, 2H), 4.86–4.91 (m, 1H), 3.85 (dd, 1H, J_1 = 4.5, J_2 = 11.5 Hz), 3.77–3.80 (m, 1H), 3.56 (dd, 1H, J_1 = 6.5, J_2 = 11.5 Hz), 3.45, (dd, 1H, J_1 = 7.0, J_2 = 11.5 Hz). ^{13}C NMR δ: 34.0, 42.7, 79.8, 115.8, 116.0, 122.4, 122.5, 144.7, 158.9, 160.9, 162.9. m/z (EI): 245, 247(M$^+$). HRMS-EI: Calcd for C$_{10}$H$_9$NOSFCl: 245.0077. Found: 245.0075.

(3-(4-Chlorophenyl)-2-(4-chlorophenylimino)thiazolidin-5-yl) methanol **(5):** White crystal. M.p. 127.0–129.7 °C.[5] IR (cm^{-1}): 1659 (C=N). ^{1}H NMR δ: 7.47–7.49 (dd, 2H, J_1 = 2.0, J_2 = 6.5 Hz), 7.31–7.33 (m, 2H), 7.20–7.26 (m, 4H), 6.09 (s, 1H), 3.96 (dd, 1H, J_1 = 6.5, J_2 = 14.5 Hz), 3.80 (dd, 1H, J_1 = 6.0, J_2 = 14.5 Hz), 3.15–3.20 (m, 1H), 2.48 (dd, 1H, J_1 = 1.0, J_2 = 6.0 Hz), 2.13 (dd, 1H, J_1 = 1.5, J_2 = 5.5 Hz). ^{13}C NMR δ: 24.2, 32.0, 55.5, 120.7 × 2, 128.3, 128.8 × 2, 130.1 × 2, 130.7 × 2, 134.7, 137.0, 139.5, 153.6. m/z (EI): 352, 354, 355(M$^+$). HRMS-EI: Calcd for C$_{16}$H$_{14}$N$_2$OSCl$_2$: 352.0204. Found: 352.0198.

(R)-*N-(5-(Chloromethyl)-1,3-oxathiolan-2-ylidene)aniline* **(3k):** Wax. ^{1}H NMR δ: 7.26–7.35 (m, 2H), 7.11–7.14 (m, 1H), 6.95–6.98 (m, 2H), 4.83–4.90 (m 1H), 3.84 (dd, 1H, J_1 = 4.4, J_2 = 11.2 Hz), 3.78 (dd, 1H, J_1 = 8.0, J_2 = 11.2 Hz), 3.53 (dd, 1H, J_1 = 6.0, J_2 = 11.2 Hz), 3.42 (dd, 1H, J_1 = 7.2, J_2 = 11.2 Hz). m/z (EI): 227, 229(M$^+$). HRMS-EI: Calcd for C$_{10}$H$_{10}$NOSCl: 227.0172. Found: 227.0173.

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