

From *Nef*-Isocyanide Adducts toward Functionalized 5-Iminothiazolidines Containing Polarized C=C Bonds

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An efficient synthesis of alkyl 5-(alkylimino)-3-aryl(alkyl)-2-(dicyanomethylene)-4-hydroxythiazolidine-4-carboxylates, containing polarized C=C bonds, *via* reaction of malononitrile, arylisothiocyanates, and the *Nef*-isocyanide adducts, under basic conditions, is described.

Introduction. – The concept of push-pull (or polarized) olefinic systems has played an important role in organic chemistry for four decades. Substitution of one C-atom of the C=C bond with electron-donating groups and of the other with electron-withdrawing groups diminishes the C=C bond order by charge separation [1–4]. The polarized structure of the C=C bond is discernible by ¹³C-NMR spectra due to the extreme deshielded position of the signal of the alkenyl C-atom on the donor side and the contrastingly shielded position of the signal of the C-atom on the acceptor side of the push-pull alkene [5].

One of the main problems of the *Nef*-isocyanide reaction is the selectivity of the nucleophilic attacks on the *Nef* adducts at either the imidoyl or the carbonyl function, which may lead to isocyanide elimination. We anticipated that the use of the α -alkoxycarbonyl substituent will render the ketone more electrophilic and probably lowers the potential elimination of isocyanide, leading to trivial acylation derivatives. This modification was found to be successful. Thus, as part of our current studies on the development of new routes to heterocyclic systems [6–9], we report herein a simple synthesis of highly functionalized 5-(alkylimino)-2-(dicyanomethylene)-4-hydroxythiazolidine derivatives containing polarized C=C bonds from the reaction of the *in situ* generated *Nef*-isocyanide adducts [10–15], malononitrile, and aryl isothiocyanate. Although, a synthesis of functionalized 2-thioxo-1,3-thiazolanes *via* reaction of primary amines, CS₂, and ethyl bromopyruvate, has been described [16], there are no published examples of 5-iminothiazolidines.

Results and Discussion. – We initiated our study with the reaction of methyl 3-[(*tert*-butyl)imino]-3-chloro-2-oxopropanoate (**3a**), obtained from methyl 2-chloro-2-oxoacetate (**1a**) and *tert*-butyl isocyanide (**2a**), with 2,2-dicyano-*N*-phenylethanethioamide potassium salt (**6a**), derived from phenyl isothiocyanate (**4a**), and malononitrile (**5**), in the presence of various bases (*Scheme 1*). The results are compiled in *Table 1*. The desired product **7a** could be obtained in 88% yield by using KOH in EtOH, in the presence of 4-Å molecular sieves (MS) (*Table 1, Entry 1*). Other bases such as

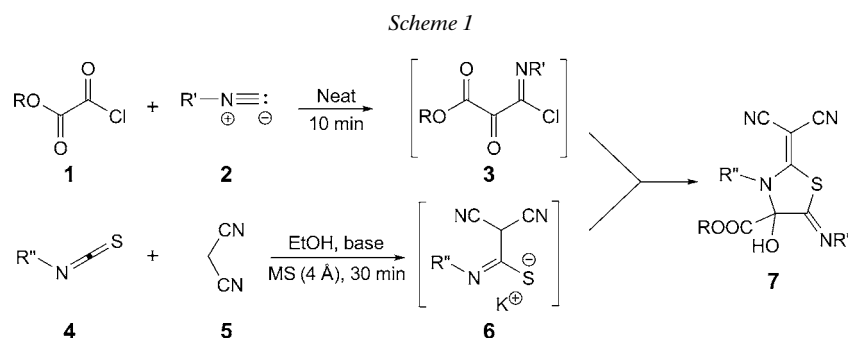


Table 1. Optimization of the Reaction Conditions for the Synthesis of Thiazolidine **7a** (R = Me, R' = ^tBu, R'' = Ph)^{a)}

Entry	Base	Time ^{b)}	Solvent	Yield [%] ^{c)}
1	KOH/Mol. sieves (MS)	30 min	EtOH	88
2	DBU ^{d)}	30 min	EtOH	78
3	Et ₃ N	1 h	EtOH	15
4	Et ₃ N	1 h	THF	12
5	EtN ⁱ Pr ₂	1 h	EtOH	35
6	DABCO ^{e)}	45 min	EtOH	62
7	K ₂ CO ₃	30 min	EtOH	57

^{a)} All reactions were carried out with 1 mmol of each substrate and base at room temperature.

^{b)} Reactions monitored by TLC. ^{c)} Yields of isolated **7a**. ^{d)} DBU = 1,8-Diazobicyclo[5.4.0]undec-7-ene.

^{e)} DABCO = 1,4-Diazobicyclo[2.2.2]octane

DBU, Et₃N, DABCO, EtNⁱPr₂, and K₂CO₃ gave lower yields of **7a** (Table 1, Entries 2–7).

Under the optimized conditions, various *Nef*-isocyanide adducts **3** and potassium salts **6** were successfully coupled to furnish the corresponding thiazolidine derivatives **7** (Table 2). The structures of 5-iminothiazolidines **7** were determined by IR, and ¹H- and ¹³C-NMR spectral data. For example, the ¹H-NMR spectrum of **7a** exhibited three *singlets* for ^tBu (1.33 ppm), MeO (3.87 ppm), and OH (5.17 ppm) H-atoms, along with characteristic *multiplets* for the aromatic H-atoms. The ¹H-decoupled ¹³C-NMR spectrum of **7a** showed 16 resonances in agreement with the proposed structure. The Ph group exhibited five and six signals in the ¹H- and ¹³C-NMR spectra, respectively. These signal patterns can be attributed not only to the presence of a stereogenic centre in **7a**, but also to a hindered rotation of the Ph residue. The polarized nature of the C=C bond in **7a** was discernible from its ¹³C-NMR shifts. The resonance of the deshielded alkenyl C-atom on the donor side was at 169.2 ppm, which is over 100 ppm at lower field compared to that of the shielded C-atom (at 58.6 ppm) on the acceptor side of this push-pull olefin. The ¹H- and ¹³C-NMR spectra of compounds **7b**–**7j** are similar to those of **7a**.

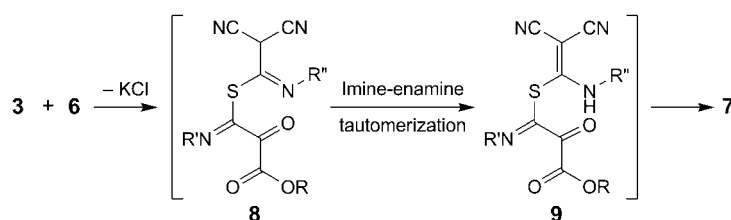
Although the mechanistic details of the formation of compounds **7** are not known, a plausible proposal is outlined in Scheme 2. The S-atom of the potassium salt **6** can

Table 2. Synthesis of Thiazolidines **7**

1 ; R	2 ; R'	4 ; R''	Product	Yield [%]
Me	^t Bu	Ph	7a	88
Me	Cyclohexyl	Ph	7b	86
Et	^t Bu	Ph	7c	91
Et	Cyclohexyl	Ph	7d	89
Et	Cyclohexyl	Et	7e	85
Me	Cyclohexyl	Et	7f	84
Et	^t Bu	4-F-C ₆ H ₄	7g	87
Et	^t Bu	2,4-Cl ₂ -C ₆ H ₃	7h	82
Me	Cyclohexyl	2,4-Cl ₂ -C ₆ H ₃	7i	76
Et	Cyclohexyl	4-NO ₂ -C ₆ H ₄	7j	89

attack on the α -ketoimidoyl chloride **3** to produce intermediate **8**, which undergoes imine-enamine tautomerization to afford **9**. This intermediate is converted to **7** by intramolecular cyclization reaction.

Scheme 2



In summary, we have described the use of malononitrile as a potential anionic nucleophile in a reaction involving isothiocyanates and *Nef*-isocyanide adducts to give highly functionalized 5-iminothiazolidines.

Experimental Part

General. All chemicals were obtained commercially and used without further purification. M.p.: *Electrothermal-9100* apparatus. IR Spectra: *Shimadzu-IR-460* spectrometer; $\tilde{\nu}$ in cm^{-1} . ¹H- and ¹³C-NMR spectra: *Bruker DRX-500 Avance* instrument at 500.1 and 125.7 MHz, resp.; δ in ppm, *J* in Hz. MS: *Finnigan-MAT-8430EI-MS* mass spectrometer; at 70 eV; in *m/z* (rel. %). Elemental analyses: *Vario EL III CHNOS* elemental analyzer.

General Procedure for the Synthesis of Compound 7. To a stirred soln. of malononitrile (**5**; 1 mmol) in 2 ml of dry EtOH and KOH (1 mmol) was added isothiocyanate (**4**, 1 mmol). After 30 min, imidoyl chloride **3**, obtained from alkyl oxalyl chloride (**1**; 1 mmol) and alkyl isocyanide (**2**, 1 mmol) at r.t. in 10 min, diluted with CH₂Cl₂ (1 ml), was added to this mixture, in the presence of 4-Å MS. The mixture was stirred for 3 h at r.t. and then concentrated under reduced pressure. The crude compound was purified by flash column chromatography (FC; hexane/AcOEt 4:1) to give the product.

Methyl 5-[tert-Butylimino]-2-(dicyanomethylidene)-4-hydroxy-3-phenyl-1,3-thiazolidine-4-carboxylate (7a). Pale-yellow powder. Yield: 0.32 g (88%). M.p. 166–168°. IR (KBr): 3192 (OH), 2211 (CN), 1761 (C=O), 1654 (C=N), 1164 (C–O). ¹H-NMR: 1.33 (s, ^tBu); 3.87 (s, MeO); 5.17 (br. s, OH); 7.19 (d, ³J = 7.8, CH); 7.45 (d, ³J = 7.6, CH); 7.47 (t, ³J = 7.9, CH); 7.51 (t, ³J = 7.8, CH); 7.56 (t, ³J = 7.7, CH). ¹³C-NMR: 28.0 (Me₃C); 53.2 (NC); 54.6 (MeO); 58.6 (C); 97.2 (C); 110.3 (CN); 114.8 (CN); 128.3

(CH); 129.6 (CH); 130.0 (CH); 131.0 (CH); 131.6 (CH); 133.4 (C); 145.6 (C); 167.1 (C); 169.2 (C). EI-MS: 370 (1, M^+), 355 (6), 311 (27), 255 (40), 227 (9), 168 (18), 100 (11), 77 (58), 57 (100). Anal. calc. for $C_{18}H_{18}N_4O_3S$ (370.43): C 58.36, H 4.90, N 15.12; found: C 57.89, H 4.84, N 15.21.

Methyl 5-(Cyclohexylimino)-2-(dicyanomethylidene)-4-hydroxy-3-phenyl-1,3-thiazolidine-4-carboxylate (7b). Red powder. Yield: 0.34 g (86%). M.p. 146–148°. IR (KBr): 3357 (OH), 2206 (CN), 1757 (C=O), 1662 (C=N), 1260 (C–O). 1H -NMR: 1.26–1.73 (*m*, 5 CH_2); 3.03–3.10 (*m*, CH); 3.71 (*s*, MeO); 5.38 (*br. s.*, OH); 7.25 (*d*, $^3J = 8.1$, CH); 7.41 (*d*, $^3J = 7.5$, CH); 7.47 (*t*, $^3J = 7.9$, CH); 7.52 (*t*, $^3J = 7.8$, CH); 7.55 (*t*, $^3J = 7.3$, CH). ^{13}C -NMR: 23.2, 24.8, 24.8, 31.5, 31.8 (5 CH_2); 50.1 (NCH); 53.6 (MeO); 68.1 (C); 97.2 (C); 110.5 (CN); 115.4 (CN); 128.3 (CH); 129.5 (CH); 129.5 (CH); 130.6 (CH); 131.0 (CH); 133.5 (C); 151.2 (C); 165.1 (C); 167.5 (C). EI-MS: 396 (1, M^+), 337 (22), 307 (11), 255 (10), 215 (40), 167 (37), 141 (14), 83 (61), 77 (100), 55 (82). Anal. calc. for $C_{20}H_{20}N_4O_3S$ (396.46): C 60.59, H 5.08, N 14.13; found: C 60.28, H 5.14, N 14.22.

Ethyl 5-[(tert-Butyl)imino]-2-(dicyanomethylidene)-4-hydroxy-3-phenyl-1,3-thiazolidine-4-carboxylate (7c). Pale-yellow powder. Yield: 0.35 g (91%). M.p. 177–179°. IR (KBr): 3203 (OH), 2208 (CN), 1762 (C=O), 1659 (C=N), 1168 (C–O). 1H -NMR: 1.33 (*t*, $^3J = 7.2$, Me); 1.33 (*s*, Me_3C); 4.22–4.25 (*m*, 1 H, CH_2O); 4.37–4.41 (*m*, 1 H, CH_2O); 5.15 (*br. s.*, OH); 7.21 (*d*, $^3J = 8.7$, CH); 7.45 (*d*, $^3J = 8.1$, CH); 7.50 (*t*, $^3J = 9.0$, CH); 7.52 (*t*, $^3J = 8.4$, CH); 7.55 (*t*, $^3J = 7.7$, CH). ^{13}C -NMR: 13.9 (Me); 28.0 (Me_3C); 52.9 (NC); 64.4 (CH_2O); 69.0 (C); 97.0 (C); 110.4 (CN); 114.8 (CN); 128.5 (CH); 129.5 (CH); 129.9 (CH); 130.9 (CH); 131.5 (CH); 133.4 (C); 145.8 (C); 166.5 (C); 169.2 (C). EI-MS: 384 (2, M^+), 311 (77), 255 (91), 201 (28), 168 (72), 116 (16), 100 (15), 77 (67), 57 (100). Anal. calc. for $C_{19}H_{20}N_4O_3S$ (384.45): C 59.36, H 5.24, N 14.57; found: C 59.02; H 5.15, N 14.50.

Ethyl 5-(Cyclohexylimino)-2-(dicyanomethylidene)-4-hydroxy-3-phenyl-1,3-thiazolidine-4-carboxylate (7d). Red powder. Yield: 0.36 g (89%). M.p. 155–157°. IR (KBr): 3422 (OH), 2350 (CN), 2208 (CN), 1760 (C=O), 1665 (C=N), 1172 (C–O). 1H -NMR: 1.16–1.76 (*m*, 5 CH_2); 1.29 (*t*, $^3J = 7.1$, Me); 2.95–2.99 (*m*, CH); 4.19–4.26 (*m*, 1 H, CH_2O); 4.34–4.41 (*m*, 1 H, CH_2O); 5.74 (*br. s.*, OH); 7.18 (*d*, $^3J = 8.2$, CH); 7.43 (*d*, $^3J = 7.6$, CH); 7.44 (*t*, $^3J = 7.4$, CH); 7.49 (*t*, $^3J = 8.3$, CH); 7.54 (*t*, $^3J = 7.3$, CH). ^{13}C -NMR: 14.0 (Me); 24.0, 24.0, 25.0, 31.9, 32.1 (5 CH_2); 53.5 (NCH); 64.6 (CH_2O); 69.2 (C); 95.8 (C); 110.4 (CN); 114.9 (CN); 128.5 (CH); 129.5 (CH); 129.9 (CH); 131.1 (CH); 131.6 (CH); 133.4 (C); 151.8 (C); 166.1 (C); 168.2 (C). EI-MS: 410 (3, M^+), 337 (94), 255 (49), 243 (29), 168 (57), 141 (13), 83 (64), 77 (51), 55 (100). Anal. calc. for $C_{21}H_{22}N_4O_3S$ (410.49): C 61.44, H 5.40, N 13.65; found: C 61.01, H 5.31, N 13.56.

Ethyl 5-(Cyclohexylimino)-2-(dicyanomethylidene)-3-ethyl-4-hydroxy-1,3-thiazolidine-4-carboxylate (7e). Yellow powder. Yield: 0.30 g (85%). M.p. 118–120°. IR (KBr): 3303 (OH), 2207 (CN), 1758 (C=O), 1664 (C=N), 1177 (C–O). 1H -NMR: 0.84–1.95 (*m*, 5 CH_2); 1.28 (*t*, $^3J = 7.1$, Me); 1.34 (*t*, $^3J = 7.2$, Me); 2.88–2.93 (*m*, CH); 3.58–3.65 (*m*, 1 H of CH_2N); 3.94–4.02 (*m*, 1 H of CH_2N); 4.22–4.29 (*m*, 1 H, CH_2O); 4.37–4.45 (*m*, 1 H, CH_2O); 5.60 (*br. s.*, OH). ^{13}C -NMR: 13.9 (Me); 14.8 (Me); 23.9, 24.0, 25.2, 31.8, 32.2 (5 CH_2); 41.2 (CH_2N); 50.8 (NCH); 64.7 (CH_2O); 69.0 (C); 95.7 (C); 113.5 (CN); 115.1 (CN); 151.3 (C); 166.8 (C); 167.0 (C). EI-MS: 362 (2, M^+), 289 (87), 207 (73), 179 (15), 92 (17), 83 (76), 55 (100). Anal. calc. for $C_{17}H_{22}N_4O_3S$ (362.45): C 56.33, H 6.12, N 15.46; found: C 56.56, H 6.19, N 15.38.

Methyl 5-(Cyclohexylimino)-2-(dicyanomethylidene)-3-ethyl-4-hydroxy-1,3-thiazolidine-4-carboxylate (7f). Yellow powder. Yield: 0.29 g (84%). M.p. 124–126°. IR (KBr): 3377 (OH), 2208 (CN), 1755 (C=O), 1663 (C=N), 1251 (C–O). 1H -NMR: 0.85–1.81 (*m*, 5 CH_2); 1.36 (*t*, $^3J = 7.1$, Me); 2.90–2.94 (*m*, CH); 3.60–3.65 (*m*, 1 H, CH_2N); 3.91 (*s*, MeO); 3.99–4.03 (*m*, 1 H, CH_2N); 5.92 (*br. s.*, OH). ^{13}C -NMR: 14.2 (Me); 23.3, 23.4, 24.6, 31.4, 31.6 (5 CH_2); 40.7 (CH_2N); 54.4 (NCH); 68.3 (MeO); 73.5 (C); 95.0 (C); 112.8 (CN); 114.4 (CN); 150.8 (C); 166.3 (C); 167.2 (C). EI-MS: 348 (2, M^+), 289 (53), 207 (41), 179 (12), 92 (25), 83 (48), 55 (100). Anal. calc. for $C_{16}H_{20}N_4O_3S$ (348.42): C 55.16, H 5.79, N 16.08; found: C 55.42, H 5.71, N 16.15.

Ethyl 5-[(tert-Butyl)imino]-2-(dicyanomethylidene)-3-(4-fluorophenyl)-4-hydroxy-1,3-thiazolidine-4-carboxylate (7g). Yellow powder. Yield: 0.35 g (87%). M.p. 146–148°. IR (KBr): 3427 (OH), 2214 (CN), 2208 (CN), 1752 (C=O), 1648 (C=N), 1187 (C–O). 1H -NMR: 1.32 (*t*, $^3J = 7.2$, Me); 1.35 (*s*, tBu); 4.22–4.25 (*m*, 1 H, CH_2O); 4.37–4.43 (*m*, 1 H, CH_2O); 5.13 (*br. s.*, OH); 7.12–7.70 (*m*, 4 CH). ^{13}C -NMR: 13.7 (Me); 28.5 (Me_3C); 58.0 (NC); 64.0 (CH_2O); 67.6 (C); 96.3 (C); 109.9 (CN); 114.1 (CN); 116.1 (*d*,

$^2J(\text{C,F}) = 23.3$, CH); 116.6 (*d*, $^2J(\text{C,F}) = 23.0$, CH); 128.8 (C); 130.0 (*d*, $^3J(\text{C,F}) = 9.2$, CH); 132.5 (*d*, $^3J(\text{C,F}) = 9.2$, CH); 149.3 (C); 162.6 (C, *d*, $^1J(\text{C,F}) = 251.0$); 165.8 (C); 169.0 (C). EI-MS: 402 (1, M^+), 329 (64), 273 (63), 217 (17), 186 (44), 167 (15), 149 (44), 116 (35), 95 (40), 71 (65), 57 (100). Anal. calc. for $\text{C}_{21}\text{H}_{21}\text{FN}_4\text{O}_3\text{S}$ (428.48): C 58.87, H 4.94, N 13.08; found: C 58.51, H 4.99, N 13.14.

Ethyl 5-[(tert-Butylimino)-3-(2,4-dichlorophenyl)-2-(dicyanomethylidene)-4-hydroxy-1,3-thiazolidine-4-carboxylate (7h). Yellow powder. Yield: 0.37 g (82%). M.p. 114–116°. IR (KBr): 3391 (OH), 2212 (CN), 1751 (C=O), 1659 (C=N), 1214 (C–O). $^1\text{H-NMR}$: 1.28 (*t*, $^3J = 7.1$, Me); 1.33 (*s*, $t\text{-Bu}$); 4.19–4.27 (*m*, 1 H, CH_2O); 4.33–4.42 (*m*, 1 H, CH_2O); 5.89 (*br. s*, OH); 7.13 (*d*, $^3J = 7.2$, CH); 7.36 (*s*, CH); 7.53 (*d*, $^3J = 7.3$, CH). $^{13}\text{C-NMR}$: 13.5 (Me); 27.5 (Me_3C); 58.1 (NC); 63.9 (CH_2O); 67.6 (C); 96.4 (C); 109.9 (CN); 114.1 (CN); 127.9 (CH); 129.9 (CH); 130.0 (CH); 130.1 (C); 132.4 (C); 136.6 (C); 146.5 (C); 165.8 (C); 169.1 (C). EI-MS: 452 (1, M^+), 423 (7), 329 (10), 217 (12), 186 (8), 167 (17), 149 (38), 83 (27), 71 (35), 57 (100). Anal. calc. for $\text{C}_{19}\text{H}_{18}\text{Cl}_2\text{N}_4\text{O}_3\text{S}$ (453.34): C 50.34, H 4.00, N 12.36; found: C 50.79, H 4.09, N 12.27.

Methyl 5-(Cyclohexylimino)-3-(2,4-dichlorophenyl)-2-(dicyanomethylidene)-4-hydroxy-1,3-thiazolidine-4-carboxylate (7i). Brown powder. Yield: 0.35 g (76%). M.p. 171–173°. IR (KBr): 3071 (OH), 2211 (CN), 1762 (C=O), 1676 (C=N), 1100 (C–O). $^1\text{H-NMR}$: 0.86–1.79 (*m*, 5 CH_2); 2.99–3.01 (*m*, CH); 3.83 (*s*, MeO); 5.84 (*br. s*, OH); 7.30 (*d*, $^3J = 8.2$, CH); 7.38 (*s*, CH); 7.50 (*d*, $^3J = 7.3$, CH). $^{13}\text{C-NMR}$: 25.3, 26.5, 30.9, 33.3, 33.4 (5 CH_2); 50.3 (NCH); 55.9 (MeO); 70.5 (C); 96.1 (C); 111.4 (CN); 115.0 (CN); 129.6 (CH); 131.9 (CH); 132.0 (CH); 132.3 (C); 134.8 (C); 139.9 (C); 152.6 (C); 167.8 (C); 168.9 (C). EI-MS: 464 (1, M^+), 445 (3), 368 (18), 335 (20), 305 (25), 238 (25), 157 (39), 98 (38), 83 (70), 67 (27), 55 (100). Anal. calc. for $\text{C}_{20}\text{H}_{18}\text{Cl}_2\text{N}_4\text{O}_3\text{S}$ (465.35): C 51.62, H 3.90, N 12.04; found: C 51.69, H 3.81, N 12.12.

Ethyl 5-(Cyclohexylimino)-2-(dicyanomethylidene)-4-hydroxy-3-(4-nitrophenyl)-1,3-thiazolidine-4-carboxylate (7j). Green powder. Yield: 0.40 g (89%). M.p. 108–110°. IR (KBr): 3420 (OH), 2215 (CN), 1758 (C=O), 1666 (C=N), 1107 (C–O). $^1\text{H-NMR}$: 1.24–1.77 (*m*, 5 CH_2); 2.96–3.00 (*m*, CH); 4.20–4.27 (*m*, 1 H, CH_2O); 4.35–4.42 (*m*, 1 H, CH_2O); 5.54 (*br. s*, OH); 7.42 (*d*, $^3J = 8.2$, CH); 7.68 (*d*, $^3J = 7.6$, CH); 8.30 (*d*, $^3J = 7.4$, CH); 8.35 (*d*, $^3J = 8.3$, CH). $^{13}\text{C-NMR}$: 14.0 (Me); 23.9, 23.9, 25.1, 32.0, 32.1 (5 CH_2); 53.7 (NCH); 65.0 (CH_2O); 69.4 (C); 95.8 (C); 110.4 (CN); 114.1 (CN); 124.8 (CH); 125.1 (CH); 129.9 (CH); 132.1 (CH); 138.8 (C); 149.1 (C); 150.6 (C); 165.8 (C); 168.3 (C). EI-MS: 455 (3, M^+), 426 (6), 273 (12), 243 (9), 226 (19), 165 (26), 138 (44), 83 (100), 69 (13), 55 (87). Anal. calc. for $\text{C}_{21}\text{H}_{21}\text{N}_5\text{O}_5\text{S}$ (455.12): C 55.37, H 4.65, N 15.38; found: C 55.29, H 4.73, N 15.30.

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