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Microwave-Assisted Coupling Reaction of *N*-Aryl Sydnones with 2-Nitromethylenethiazolidine: Unexpected Formation of (*Z*)-2-(Nitro((*E*)-*p*-substitutedphenyldiazenyl)methylene)thiazolidines

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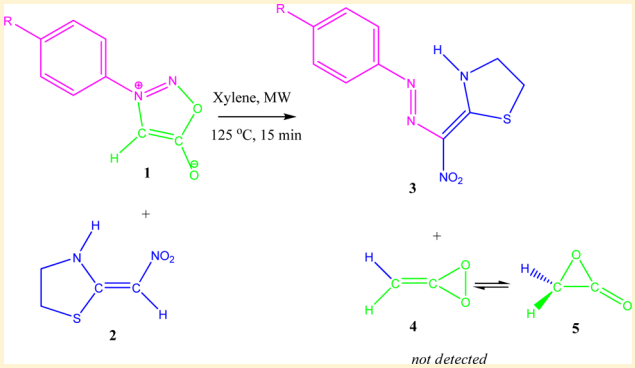
Supporting Information

ABSTRACT: Reaction of *N*-aryl sydnones with 2-nitromethylenethiazolidine straightforwardly gives rise to the formation of (*Z*)-2-(nitro((*E*)-*p*-substitutedphenyldiazenyl)methylene)thiazolidines in xylene and dimethoxyethane under microwave irradiation. A meaningful and plausible mechanism for this transformation is proposed, which anticipates the extrusion of an aceto-lactone-like moiety before a coupling occurs. The structures of all the new compounds were identified on the basis of the data obtained from the NMR, IR, X-ray diffraction spectra, HRMS measurements, and physical characteristics.



Sydnones constitute a well-defined class of mesoionic compounds obtained by the action of acetic anhydride or 1,3-dibromo-5,5-dimethylhydantoin on *N*-nitroso derivative.[1,2] These compounds are of interest because of the varied types of biological activity displayed by some of them, particularly sydnone 4-heterocycles.[3−5] On the other hand, thiazolidine ring substituted heterocyclic scaffolds have been reported as insecticide agent against Lepidoptera species.[6−8] Diazenyl substituted compounds have also been found to exhibit various biological activities such as antifungal, antibacterial, and anticancer activities.[9−11]

Taking account of the above considerations, plus our continuous interest in 1,3-dipolar cycloaddition chemistry of various ylides leading to pyrazoles, triazoles and spiropyrroles,[12−15] and our recent interest in 2-nitromethylene thiazolidine chemistry,[16−19] we have focused herein on the reaction of *N*-aryl sydnones as ylide generating precursor with 2-nitromethylenethiazolidine as dipolarophilic reagent.

Syndones are known to react with dipolarophiles in 1,3-dipolar cycloaddition route.[20,21] But, however, in our case, mesoionic sydnones **1** underwent, unexpectedly, a coupling reaction with the electron deficient dipolarophile, 2-nitromethylene thiazolidine **2**, under microwave irradiation instead of 1,3-dipolar cycloaddition and afforded 8 novel (*Z*)-2-(nitro((*E*)-*p*-substitutedphenyldiazenyl)methylene)-thiazolidines **3a−h** (Scheme 1), and their structures were identified by means of IR, NMR, mass spectra, X-ray diffraction data and physical characteristics. We were able to crystallize one of the compounds (**3b**) to obtain X-ray diffraction data leading us to establish exact structures of the end products (see Supporting Information).

Upon an initial examination of the structures of the reaction products, basically, on the basis of NMR, IR and HRMS data, despite the expected spiro structures (Scheme 2) we concluded that fused cyclic compounds (Scheme 3) might be formed. This could be confirmed by the existing two triplets of the methylene protons, which are originated from the 2-nitromethylenethiazolidine. In addition, NH proton, which is located adjacent to nitro group, might arise quite much deshielded at around 14.5 ppm in the proton NMR spectra and exhibits a strong absorption at around 3300−3400 cm^{-1} in the IR spectra. Also, LC−MS spectra and HRMS measurements reveal that M + H values exactly coincide with the molecular formulas of the structures. But, however, when we were able to obtain a fine crystal of the compound **3b**, X-ray diffraction ORTEP view revealed the structures assigned as depicted in Scheme 1. As for ^{13}C NMR signals, the carbon resonance of the one attached to nitro group appeared at around 162 ppm, and the other exocyclic double bond carbon did at around 150 ppm (in DMSO-d_6). We have noticed that one carbon resonance is missing for some samples recorded in $CDCl_3$. For this reason, we decided to record ^{13}C NMR spectra in dimethyl sulfoxide-d_6 instead of $CDCl_3$ for those samples. In this regard, we observed 8 carbon signals for compound **3g** and 9 carbons for **3b**. NH proton chemical shifts have arose at around 12.7 ppm in DMSO-d_6.

A possible reaction mechanism for this transformation was proposed below (Scheme 4). It seems to be most likely that a structure resembling acetolactone or methylenedioxirane[22−26]

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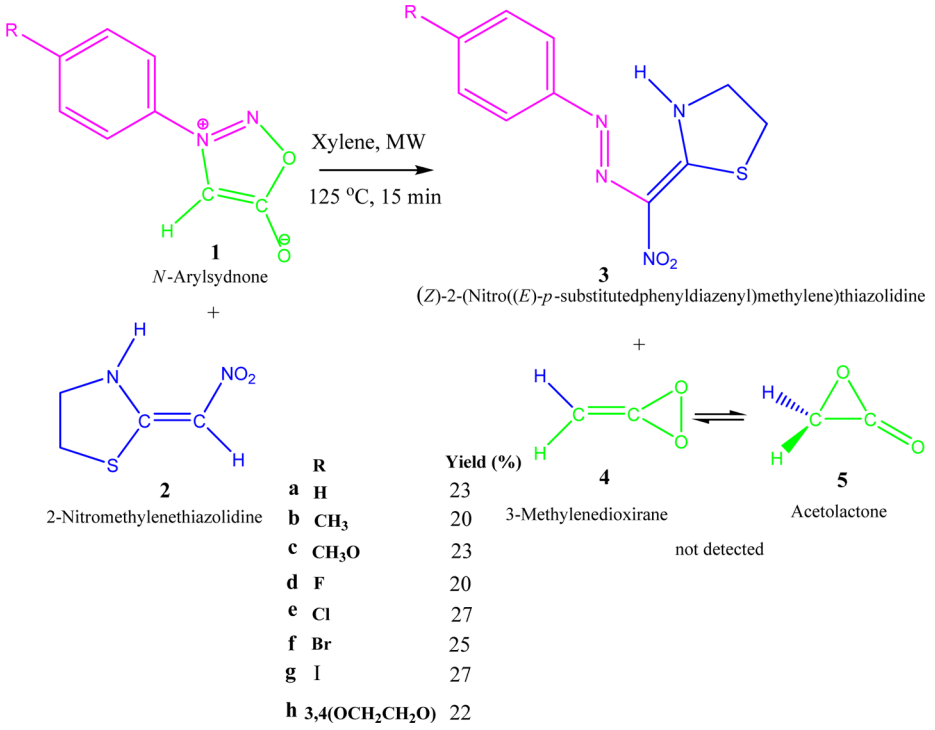
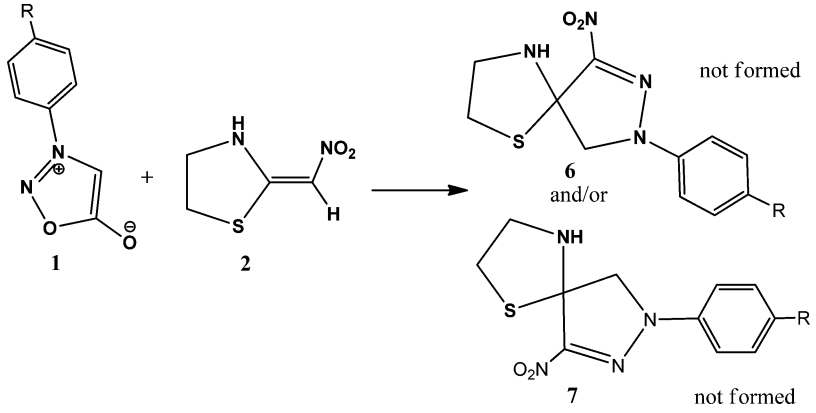
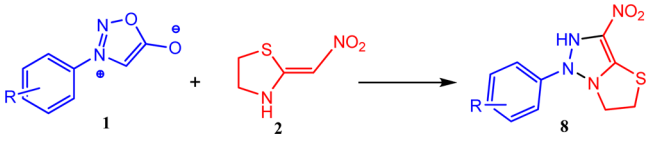
Scheme 1. Reaction between *N*-Aryl Sydnones 1 and 2-Nitromethylenethiazolidine 2



Scheme 2. Anticipated Reaction Products of Sydnone-2-nitromethylenethiazolidine 1,3-Dipolar Cycloaddition Leading to Spiro Heterocycles 6 or 7



Scheme 3. Anticipated Reaction Products of Sydnone-2-nitromethylenethiazolidine 1,3-Dipolar Cycloaddition Leading to Fused Heterocycles 8



is released during the reaction but not isolated (Scheme 5). It was reported that only in the case of trifluoromethyl substitution, these structures are stable and can be isolated. In addition, in a study it was reported that during the nitrosation of amino acids, lactones have been formed as alkylating agents.[27]

Schafer and Gewald reported that *N*,*N*′-disubstituted 1-nitro-2,2-diamino ethylenes underwent an azo-coupling with benzene diazonium chlorides to form 1-nitro-1-arylazo-2,2-diamino ethylene (Scheme 6).[28]

NH proton shifted up to 14.5 ppm was not frequently encountered. Therefore, we may think about a tautomeric equilibriation in NMR solvent ($CDCl_3$) or a resonance form as depicted in Scheme 7. In this way, due to being closer to quite electron deficient nitrogens via a complete electron delocalization, we can consider that OH proton arises at around 14.5 ppm which shifts to 12.7 in DMSO-d_6. Another supporting evidence might be our failed attempts of trying D_2O exchange of NH proton, caused by this fast equilibrium.

In conclusion, this work represents, to the best of our knowledge, the first example of the *N*-aryl sydnone-2-nitromethylenethiazolidine reaction resembling a diazo coupling at which possibly an intermediate anticipated as acetolactone (oxiranone) or methylenedioxirane is liberated and so far not reported.

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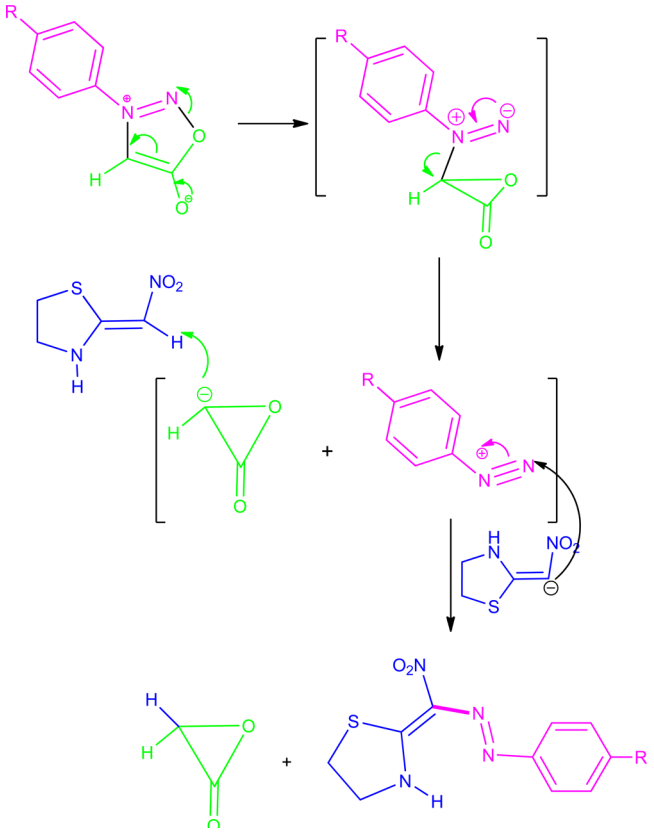
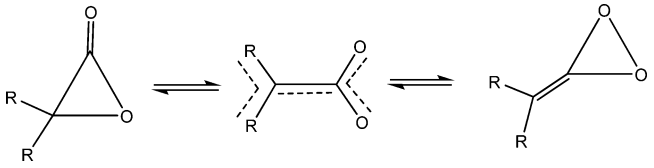
Scheme 4. Proposed Mechanism for the Generation of 3a−h



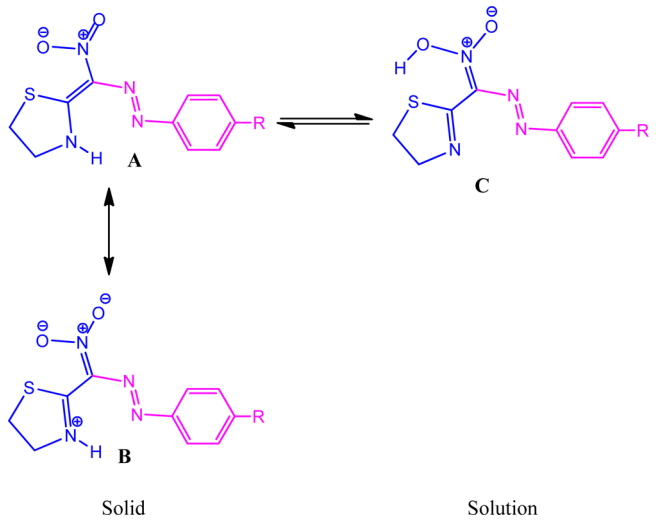
Scheme 5. Resonance Forms of Acetolactone (Oxiranone) Structure



EXPERIMENTAL SECTION

General Methods. Reagents were purchased from commercial sources and were used as received. CEM DISCOVER Microwave Reactor was used and progress of the reactions were monitored by TLC. ^{1}H NMR and ^{13}C NMR (400 and 100 MHz, respectively) spectra were recorded in $CDCl_3$ and DMSO-d_6. ^{1}H NMR chemical shifts are reported in parts per million relative to tetramethylsilane (TMS) with the solvent resonance employed as the internal standard ($CDCl_3$ at 7.26 ppm, DMSO-d_6 at 2.50 ppm). Data are reported as follows: chemical shift (multiplicity, coupling constant(s) in Hz, integration). Multiplicities are abbreviated as follows: s = singlet, br s = broad singlet, d = doublet, t = triplet, q = quartet, m = multiplet. ^{13}C

Scheme 7. Nitro-aci-nitrolic Acid Type Tautomerization and Resonance Form of (*Z*)-2-(Nitro((*E*)-*p*-substitutedphenyldiazenyl)methylene) Thiazolidines



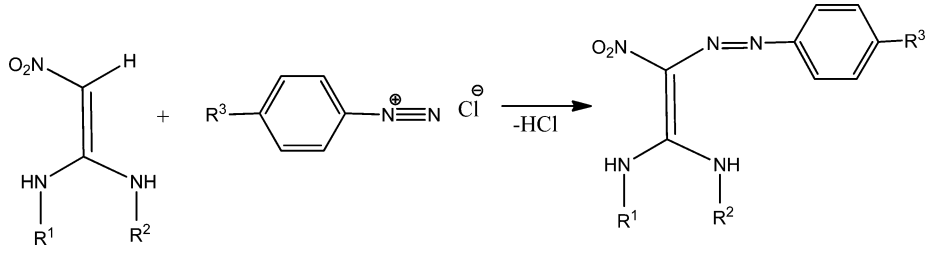
NMR chemical shifts are reported in parts per million from tetramethylsilane (TMS) with the solvent resonance as the internal standard ($CDCl_3$ at 77.20 ppm, DMSO-d_6 at 39.51 ppm). IR spectra were recorded in KBr; $\tilde{\nu}$ in cm^{-1}. Mass spectra were run on an LC−MS instrument equipped with an ESI probe operating in positive or negative ion mode, in m/z (rel. %). HRMS was performed on a TOF LC−MS equipped with an ESI probe operating in positive or negative ion mode.

General Procedure for Synthesis of (*Z*)-2-(Nitro((*E*)-*p*-substitutedphenyldiazenyl)methylene)thiazolidines 3a−h. A mixture of *p*-substitutedphenyl sydnone **1** (0.5 mmol) and 2-nitromethylenethiazolidine **2** (0.5 mmol, 73 mg) in xylene (5 mL) was irradiated by microwave (CEM Discover) at 125 °C, which was measured by internal probe, for 15 min. The reaction was monitored by TLC (*n*-hexane/EtOAc,1:1). After the completion of the reaction, the solvent was removed under the reduced pressure and crude product was chromatographed to give **3a−h**.

(Z)-2-(Nitro((E)-phenyldiazenyl)methylene)thiazolidine ***(3a)***. Yield: 28.7 mg, 52%. Yellow oil: R_f 0.38 (EtOAc:*n*-hexane, 1:1); IR (KBr, cm^{-1}) 3419 (NH), 2926, 1631, 1554, 1263, 1049, 794; ^{1}H NMR (400 MHz, $CDCl_3$) δ 14.56 (s, 1H, NH), 7.51 (dd, J = 7.6, 1.2 Hz, 2H), 7.41 (t, J = 8.2 Hz, 2H), 7.25−7.21 (m, 1H), 4.46 (t, J = 8.6 Hz, 2H, NCH_2), 3.24 (t, J = 8.8 Hz, 2H, SCH_2); ^{13}C NMR (126 MHz, $CDCl_3$) δ 162.6, 142.6, 129.8, 126.8, 117.7, 61.5 (NCH_2), 29.8 (SCH_2); LC−MS (AP-ESI) m/z (%) 251.7 [M^+ + H]; HRMS m/z (ESI-TOF, [M + H]$^+$) calcd for $C_{10}H_{11}N_4O_2S$ 251.0603, found 251.0589.

(Z)-2-(Nitro((E)-p-tolyldiazenyl)methylene)thiazolidine ***(3b)***. Light yellow needles (dichloromethane:*n*-hexane). Yield 26.4 mg, 20%: mp 135 °C (decomposed); R_f 0.38 (EtOAc:*n*-hexane, 1:1) IR (KBr, cm^{-1}) 3356 (NH), 2920, 1653, 1548, 1421, 1276, 1053, 817, 734; ^{1}H NMR (400 MHz, $CDCl_3$) δ 14.55 (s, 1H, NH), 7.42 (d, J = 8.0 Hz, 2H),

Scheme 6. Azo Coupling of 1-Nitro-2,2-diamino Ethylenes



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7.21 (d, J = 8.4 Hz, 2H), 4.42 (t, J = 8.6 Hz, 2H, NCH_2), 3.21 (t, J = 8.4 Hz, 2H, SCH_2), 2.35 (s, 3H); ^{1}H NMR (400 MHz, DMSO-d_6) δ 12.44 (br s, 1H, NH), 7.63 (d, J = 8.0 Hz, 2H), 7.24 (d, J = 8.4 Hz, 2H), 4.16 (t, J = 8.6 Hz, 2H, NCH_2), 3.25 (t, J = 8.4 Hz, 2H, SCH_2), 2.30 (s, 3H).^{13}C NMR (100 MHz, $CDCl_3$) δ 162.5 (C=**C**-NO_2), 140.6, 137.0, 130.2, 117.8, 60.8 (NCH_2), 30.5 (SCH_2), 21.2 (CH_3); ^{13}C NMR (100 MHz, DMSO-d_6) δ 162.7 (C=**C**-NO_2), 148.1 (**C**=C-NO_2), 138.7, 135.8, 130.3, 121.1, 53.9 (NCH_2), 29.2 (SCH_2), 21.3 (CH_3).LC−MS (AP-ESI) m/z (%) 265.7 [M^+ + H]; HRMS m/z (ESI-TOF, [M + H]$^+$) calcd for $C_{11}H_{13}N_4O_2S$ 265.0759, found 265.0750.

(Z)-2-(((E)-(4-Methoxyphenyl)diazenyl)(nitro)methylene)-thiazolidine **(3c)**. Orange oil. Yield 32.2 mg, 23%: R_f 0.36 (EtOAc:*n*-hexane, 1:1); IR (KBr, cm^{-1}) 3423 (NH), 2916, 1664, 1514, 1467, 1379, 1263, 738; ^{1}H NMR (400 MHz, $CDCl_3$) δ 14.24 (s, 1H, NH), 7.52 (d, J = 8.8 Hz, 2H), 6.94 (d, J = 8.8 Hz, 2H), 4.38 (t, J = 8.6 Hz, 2H, NCH_2), 3.84 (s, 3H, OCH_3), 3.23 (t, J = 8.6 Hz, 2H, SCH_2); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.7 (C=**C**-NO_2), 159.2 (**C**−$OCHH_3$), 138.6, 135.2, 120.2, 114.8, 58.5 (NCH_2), 55.6 (OCH_3), 30.1 (SCH_2); LC−MS (AP-ESI) m/z (%) 281 [M^+ + H]; HRMS m/z (ESI-TOF, [M + H]$^+$) calcd for $C_{11}H_{13}N_4O_3S$:281.0708, found 281.0701.

(Z)-2-(((E)-(4-Fluorophenyl)diazenyl)(nitro)methylene)-thiazolidine **(3d)**. Orange oil. Yield 26.8 mg, 20%: R_f 0.35 (EtOAc:*n*-hexane, 1:1); IR (KBr, cm^{-1}) 3437 (NH), 2916, 1637, 1548, 1483, 1427, 1267, 839, 734; ^{1}H NMR (400 MHz, $CDCl_3$) δ 14.24 (s, 1H, NH), 7.55−7.52 (m, 2H), 7.12 (t, J = 8.6 Hz, 2H), 4.41 (t, J = 8.8 Hz, 2H), 3.25 (t, J = 8.6 Hz, 2H); ^{13}C NMR (126 MHz, $CDCl_3$) δ 163.0 (C=**C**-NO_2), 161.6 (C−F, d, J = 245.8 Hz), 140.7, 134.8, 120.1, 116.8, 59.5 (NCH_2), 30.5 (SCH_2); LC−MS (AP-ESI) m/z (%) 269 [M^+ + H]; HRMS m/z (ESI-TOF, [M + H]$^+$) calcd for $C_{10}H_{10}N_4O_2FS$ 269.0509, found 269.0497.

(Z)-2-(((E)-(4-Chlorophenyl)diazenyl)(nitro)methylene)-thiazolidine **(3e)**. Yellow oil. Yield 38.3 mg, 27%: R_f 0.35 (EtOAc:*n*-hexane, 1:1); IR (KBr, cm^{-1}) 3419 (NH), 2916, 1629, 1548, 1483, 1419, 1267, 835, 732; ^{1}H NMR (400 MHz, $CDCl_3$) δ 14.34 (s, 1H, NH), 7.47 (d, J = 9.2 Hz, 2H), 7.38 (d, J = 8.4 Hz, 2H), 4.43 (t, J = 8.8 Hz, 2H, NCH_2), 3.25 (t, J = 8.4 Hz, 2H, SCH_2); ^{13}C NMR (126 MHz, $CDCl_3$) δ 162.7 (C=**C**-NO_2), 142.1, 132.1, 129.7, 119.1, 60.3 (NCH_2), 30.6 (SCH_2); LC−MS (AP-ESI) m/z (%) 285.1 [M^+ + H]; HRMS m/z (ESI-TOF, [M + H]$^+$) calcd for $C_{10}H_{10}N_4O_2SCl$ 285.0213, found 285.0200.

(Z)-2-(((E)-(4-Bromophenyl)diazenyl)(nitro)methylene)-thiazolidine **(3f)**. Yellow oil. Yield 41.0 mg, 25%: R_f 0.34 (EtOAc:*n*-hexane, 1:1); IR (KBr, cm^{-1}) 3423 (NH), 2926, 1743, 1550, 1492, 1265, 1051, 829, 738; ^{1}H NMR (400 MHz, $CDCl_3$) δ 14.36 (s, 1H, NH), 7.53 (d, J = 8.8 Hz, 2H), 7.41 (d, J = 8.8 Hz, 2H), 4.43 (t, J = 8.8 Hz, 2H, NCH_2), 3.25 (t, J = 8.8 Hz, 2H, SCH_2); ^{13}C NMR (100 MHz, $CDCl_3$) δ 165.4 (C=**C**-NO_2), 150.5 (**C**=C-NO_2), 132.8, 129.9, 129.2, 125.3, 66.7 (NCH_2), 33.1 (SCH_2); LC−MS (AP-ESI) m/z (%) 329 [M^+ + H]; HRMS m/z (ESI-TOF, [M + H]$^+$) calcd for $C_{10}H_{10}N_4O_2S^{79}Br$ 328.9708, found 328.9685.

(Z)-2-(((E)-(4-Iodophenyl)diazenyl)(nitro)methylene)thiazolidine **(3g)**. Yellow oil. Yield 50.6 mg, 27%: R_f 0.36 (EtOAc:*n*-hexane, 1:1); IR (KBr, cm^{-1}) 3323 (NH), 2924, 1663, 1546, 1485, 1267, 1051, 1003, 823, 738; ^{1}H NMR (400 MHz, $CDCl_3$) δ 14.39 (s, 1H, NH), 7.72 (d, J = 8.8 Hz, 2H), 7.27 (d, J = 10.0 Hz, 2H), 4.43 (t, J = 8.6 Hz, 2H, NCH_2), 3.25 (t, J = 8.6 Hz, 2H, SCH_2); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.8, 143.0, 138.6, 119.6, 90.9 (C−I), 60.4 (NCH_2), 30.6 (SCH_2); ^{13}C NMR (100 MHz, DMSO-d_6) δ 163.0 (C=**C**-NO_2), 150.2 (**C**=C-NO_2), 138.6, 136.1, 123.2, 94.9 (C−I), 53.7 (NCH_2), 29.9 (SCH_2); LC−MS (AP-ESI) m/z (%) 377.0 [M^+ + H]; HRMS m/z (ESI-TOF, [M + H]$^+$) calcd for $C_{10}H_{10}N_4O_2SI$ 376.9569, found 376.9570.

(Z)-2-(((E)-Benzo[d][1,3]dioxol-5-yldiazenyl)(nitro)methylene)-thiazolidine **(3h)**. Brown oil. Yield 32.3 mg, 22%. R_f 0.32 (EtOAc:*n*-hexane, 1:1); IR (KBr, cm^{-1}) 3319 (NH), 2924, 1689, 1546, 1491, 1253, 1035, 929, 810, 742; ^{1}H NMR (400 MHz, $CDCl_3$) δ 14.02 (s, 1H, NH), 7.29 (d, J = 8.8 Hz, 1H), 7.01 (dd, J = 8.4, 2.0 Hz, 1H), 6.99 (dd, J = 8.0, 2.0 Hz, 1H), 6.04 (s, 2H, OCH_2O), 4.37 (t, J = 8.4 Hz, 2H, NCH_2), 3.26 (t, J = 8.4 Hz, 2H, SCH_2); ^{13}C NMR (100 MHz, $CDCl_3$) δ 162.9 (C=**C**-NO_2), 149.1 (**C**=C-NO_2), 147.4, 140.9, 114.5, 108.3, 101.9, 98.9 (OCH_2O), 57.8 (NCH_2), 30.0 (SCH_2); LC−MS (AP-ESI) m/z (%) 295 [M + H]; HRMS m/z (ESI-TOF, [M + H]$^+$) calcd for $C_{11}H_{11}N_4O_4S$ 295.0501, found 295.0490.

■ ASSOCIATED CONTENT

Ⓢ Supporting Information

X-ray ORTEP view of the compound **3b**, ^{1}H, ^{13}C NMR, COSY and NOESY of the compound **3h**, HRMS spectra of all new compounds. This material is available free of charge via the Internet at http://pubs.acs.org. X-ray diffraction data of compound **3b** have been deposited at the Cambridge Crystallographic Data Centre and assigned CCDC 996089 deposition number. It can be obtained free of charge from http://www.ccdc.cam.ac.uk/data_request/cif.

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Notes

The authors declare no competing financial interest.

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■ REFERENCES

(1) Earl, J. C.; Mackney, A. W. *J. Chem. Soc.* **1935**, 899.
(2) Azarifar, D.; Bosra, H. G.; Zolfigol, M.-A.; Tajbaksh, M. *Heterocycles* **2006**, *68*, 175.
(3) Ollis, W. D.; Ramsden, C. A. In *Advances in Heterocyclic Chemistry;* Katritzky, A. R., Boulton, A. J., Eds.; Elsevier: Amsterdam, 1976; Vol. *19*, pp 1−122.
(4) Takayuki, N.; Susumu, N.; Kiyoshi, T.; Kenji, M.; Kei-Ichi, F.; Hiroshi, K. *J. Antibiot.* **1968**, *21*, 290.
(5) Upadhya, K. G.; Badami, B. V.; Puranik, G. S. *Arch. Pharm. (Weinheim, Ger.)* **1980**, *313*, 684.
(6) Shiokawa, K.; Moriya, K.; Shibuya, K.; Hattori, Y.; Tsuboi, S.; Kagabu, S. *Biosci., Biotechnol., Biochem.* **1992**, *56*, 1364.
(7) Nishida, S.; Maruoka, H.; Yoshimura, Y.; Goto, T.; Tomita, R.; Masumoto, E.; Okabe, F.; Yamagata, K.; Fujioka, T. *J. Heterocycl. Chem.* **2012**, *49*, 303.
(8) Liu, K.; Rao, W.; Parikh, H.; Li, Q. B.; Guo, T. L.; Grant, S.; Kellogg, G. E.; Zhang, S. J. *Eur. J. Med. Chem.* **2012**, *47*, 125.
(9) Bae, S. J.; Ha, Y. M.; Kim, J. A.; Park, J. Y.; Ha, T. K.; Park, D.; Chun, P.; Park, N. H.; Moon, H. R.; Chung, H. Y. *Biosci., Biotechnol., Biochem.* **2013**, *77*, 65.
(10) Chopde, H. N.; Pagadala, R.; Meshram, J. S.; Jetti, V. *J. Heterocycl. Chem.* **2011**, *48*, 1323.
(11) Baul, T. S. B.; Paul, A.; Pellerito, L.; Scopelliti, M.; Duthie, A.; De Vos, D.; Verma, R. P.; Englerf, U. *J. Inorg. Biochem.* **2012**, *107*, 119.
(12) Yıldırım, M.; Dürüst, Y. *Tetrahedron* **2011**, *67*, 3209.
(13) Dürüst, Y.; Yıldırım, M.; Fronczek, C. F.; Fronczek, F. R. *Monatsh. Chem.* **2012**, *143*, 127.
(14) Dürüst, Y.; Sağırlı, A.; Fronczek, F. R. *Mol. Diversity* **2011**, *15*, 799.
(15) Dürüst, Y.; Karakuş, H.; Kaiser, M.; Tasdemir, D. *Eur. J. Med. Chem.* **2012**, *48*, 296.
(16) Yıldırım, M.; Çelikel, D.; Dürüst, Y.; Knight, D. W.; Kariuki, B. M. *Tetrahedron* **2014**, *70*, 2122.
(17) Altuğ, C.; Burnett, A. K.; Caner, E.; Dürüst, Y.; Elliott, M. C.; Glanville, R. P. J.; Guy, C.; Westwell, A. D. *Tetrahedron* **2011**, *67*, 9522.

(18) Altuğ, C.; Dürüst, Y.; Elliott, M. C. *Tetrahedron Lett.* **2009**, *50*, 7392.
(19) Altuğ, C.; Dürüst, Y.; Elliott, M. C.; Kariuki, B. M. *Tetrahedron Lett.* **2009**, *50*, 4919.
(20) Gribble, G. W. Mesoionic Ring Systems. In *Synthetic Applications of 1,3-Dipolar Cycloaddition Chemistry Toward Heterocycles and Natural Products*; Padwa, A., Pearson, W. H., Eds.; Wiley & Sons: Hoboken, NJ, 2003; pp 681−753.
(21) Browne, D. L.; Harrity, J. P. A. *Tetrahedron* **2010**, *66*, 553.
(22) Liebman, J. F.; Greenberg, A. *J. Org. Chem.* **1974**, *39*, 123.
(23) Ruggiero, G. D.; Williams, I. H. *J. Chem. Soc., Perkin Trans.* **2001**, *2*, 733.
(24) Rodriquez, C. F.; Williams, I. H. *J. Chem. Soc., Perkin Trans.* **1997**, *2*, 953.
(25) Chung, C. S. C. *J. Mol. Struct.* **1976**, *30*, 189.
(26) Bach, R. D.; Dmitrenko, O. *J. Am. Chem. Soc.* **2006**, *128*, 4598.
(27) Garcia-Santos, M. D. P.; Calle, E.; Casado, J. *J. Am. Chem. Soc.* **2001**, *123*, 7506.
(28) Schafer, H.; Gewald, K. *J. Prakt. Chem.* **1980**, *322*, 87.

dx.doi.org/10.1021/jo5010229 | *J. Org. Chem.* 2014, 79, 6380−6384