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ORIGINAL PAPER

Synthesis and Crystal Structure Determination of Methyl 2-acetyl-5′-phenyl-2*H*-spiro[benzo[*d*]isothiazole-3,3′-pyrazole]-1,1-dioxide-2′(4′*H*)-carboxylate and Methyl 2-acetyl-5′-(2-thienyl)-2*H*-spiro[benzo[*d*]isothiazole-3,3′-pyrazole]-1,1-dioxide-2′(4′*H*)-carboxylate

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Abstract Dilithiated *C*(α), *N*-carbomethoxyhydrazones were condensed with lithiated methyl 2-(aminosulfonyl)-benzoate to afford intermediates that were isolated and not characterized but cyclized with acetic anhydride, which also resulted in *N*-acetylation. The X-ray crystal structure determinations of methyl 2-acetyl-5′-phenyl-2*H*-spiro[benzo[*d*]isothiazole-3,3′-pyrazole]-1,1-dioxide-2′(4′*H*)-carboxylate and methyl 2-acetyl-5′-(2-thienyl)-2*H*-spiro[benzo[*d*]isothiazole-3,3′-pyrazole]-1,1-dioxide-2′(4′*H*)-carboxylate products were a follow up for absorption spectra, and they confirmed their structures. Mechanistic intermediates to describe the reaction may include *C*-acylated intermediates that cyclize to spiro(*N*-benzoisothiazole dioxide-pyrazole) instead of *N*-carbomethoxypyrazole-*ortho*-benzenesulfonamides. Crystals of $C_{19}H_{17}N_3O_5S$ **7** are monoclinic, $P2_1/c$, $a = 11.899(2)$ Å, $b = 17.562(4)$ Å, $c = 9.484(2)$ Å, $\beta = 111.03(3)°$, $Z = 4$, $V = 1849.9(6)$ Å^3, $R_1 = 0.0857$ and $wR_2 = 0.2216$ for reflections with $I > 2\sigma(I)$; crystals of $C_{17}H_{15}N_3O_5S_2$ **8** are orthorhombic, $Pbca$, $a = 16.045(3)$ Å, $b = 10.746(2)$ Å, $c = 20.389(4)$ Å, $Z = 8$, $V = 3516(1)$ Å^3, $R_1 = 0.0841$ and $wR_2 = 0.2179$ for all reflections with $I > 2\sigma(I)$.

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Introduction

Pyrazoles have been prepared by several methods, and they have been used as synthetic intermediates for the preparation of other compounds, such as select ketones [1], for spectral studies recently including solid-state NMR spectra of pyrazolylborate complexes [2–5], for theoretical investigations recently including the use of chemical shifts versus coupling constants for studying tautomerism [6–8], for their biological potential in medicine which has recently been reviewed [9, 10] and in agriculture with an impressive number of patent citations (e.g., insecticides, plant growth enhancers) [11, 12]. Two of the best documented preparative methods [13–15] involve the condensation–cyclization of β-dicarbonyl compounds with substituted hydrazines, and the 1,3-dipolar addition of nitrilimines with alkynes or alkenes.

Pyrazoles have also been a part of a spiro-heterocyclic system with the spiro carbon atom between the pyrazole ring and other heterocyclic rings, with the structures of some of them being supported by X-ray analysis [16–23]. Examples include spirocyclic oxindole derivatives of an aminopyran condensed with the pyrazolic nucleus [16]; spiro-fused azirino-pyrazolones, a new heterocyclic system [18]; regioselective 1,3-dipolar cycloaddition to afford spiro(pyrazoline-chromanones) [19], or spiro(benzothiepine-pyrazol)-ones [20], or bis-spiro(pyrazoline-chroman (thiochroman)-one derivatives employing bis-nitrilimines [21], or spiro-fused-isoxazolino-pyrazolones; oxa-triazaspiro-

dienones; and a pyrazoline derivative of eunicin acetate, a lengthy name spiro compound [22].

Benzoisothiazole dioxides, (1,2-benzoisothiazole 1,1-dioxides) (BIDs), have received investigation regarding their synthesis and uses, with a few reports where BIDs have also been a part of a spiro-heterocyclic system, and one supported by an X-ray study [24, 25]. There are no examples of spiro(BID-pyrazoles).

A developing synthetic method for pyrazoles from this laboratory involves the condensation-cyclization of numerous polylithiated hydrazones with select electrophilic reagents, usually esters. In addition to finding the general reaction parameters for affecting the synthesis of a particular pyrazole, challenges arise when choosing the substituted hydrazone along with a satisfactory electrophilic reagent. In the past trilithiated hydrazones, dilithiated phenylhydrazones, and dilithiated carboalkoxyhydrazones have been investigated along with their condensation–cyclization with a variety of esters, many of them being straightforward [26] and others presenting a substantial challenge [27]. On occasion, an unexpected reaction occurred, and this led to new products plus additional investigations [28].

Methyl 2-(aminosulfonyl)benzoate **5** has been the type of electrophilic reagent used by us that has given rise to several types of heterocyclic compounds: pyrazoles [27] and spiro(benzisothiazole-isoxazole)dioxides [24]. For example, dilithiated phenylhydrazones underwent condensation–cyclization with lithiated methyl 2-(aminosulfonyl)benzoate **6** or saccharin lithium to afford 2-(1-phenyl-1*H*-pyrazol-5-yl)benzenesulfonamides [27]; however, when dilithiated oximes were treated with this ester-sulfonamide **5**, spiro[benzoisothiazole-isoxazole]dioxides [24] resulted instead of isoxazole-*ortho*-benzenesulfonamides. Another investigation involved the condensation-cyclization-hydrolysis-decarboxylation of dilithiated *N*-carbo-*tert*-butoxyhydrazones with this ester-sulfonamide **5** to afford *NH*-pyrazolyl-*ortho*-benzenesulfonamides [29].

The last synthesis indicated the possibility of success for condensation–cyclization of *N*-carbomethoxyhydrazones **3** with this ester-sulfonamide **5** to form *C*-acylated intermediates with potential, although limited, for cyclization to the *N*-carbomethoxypyrazole-*ortho*-benzenesulfonamides. The difficulty envisioned for cyclization of *C*-acylated intermediates could result from challenging resonance, inductive and/or steric effects of the ester-sulfonamide moiety.

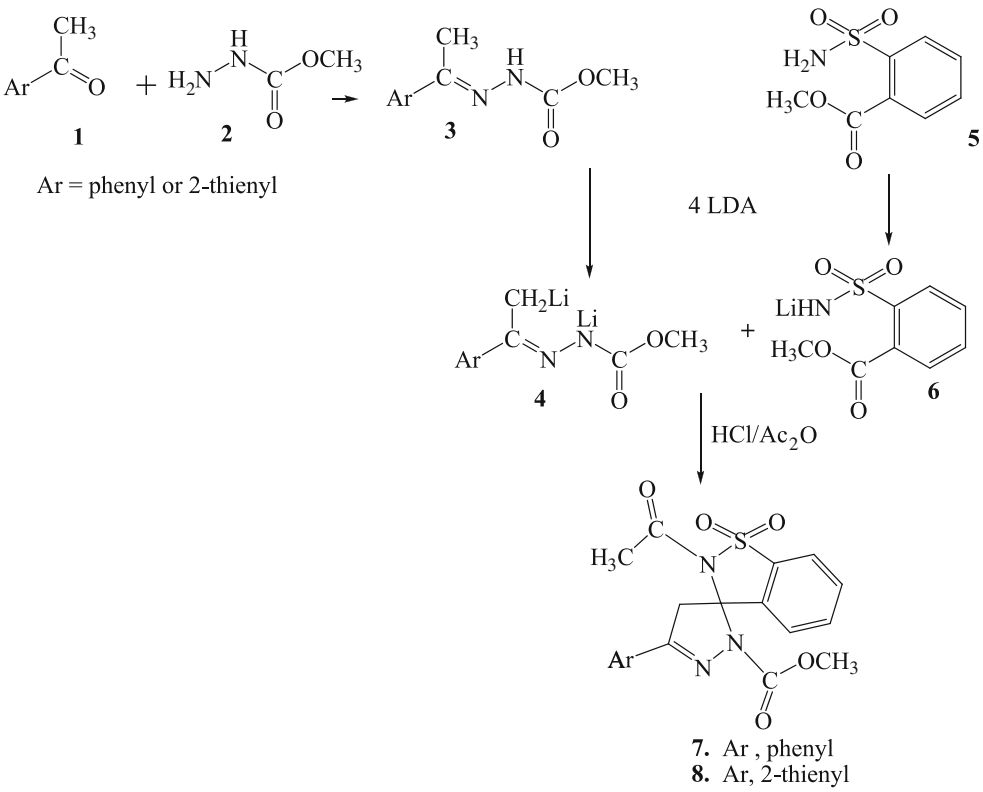
Experimental Section: Synthesis

Materials and Characterization

Fourier transform infrared spectra were obtained with a Nicolet Impact 410 FT-IR. Proton and ^{13}C NMR spectra were obtained with a Varian Associates Mercury Oxford 300 MHz nuclear magnetic resonance spectrometer, and chemical shifts were recorded in ppm downfield from an internal tetramethylsilane standard. Combustion analyses were performed by Quantitative Technologies, Inc., P.O. Box 470, Whitehouse, NJ 08888. LCMS analyses were measured on a Thermo-Finnigan LCQ Advantage system with the Surveyor autosampler, Surveyor pump, and LCQ Advantage Max mass spectral detector using electrospray ionization; 2 mg samples were prepared in 2 mL/L of acetonitrile; 10 μL injections were pumped at 1.00 mL/min isocratically with 70% acetonitrile and 30% water, each buffered with 0.1% formic acid by volume; 15 min runs were reproduced in both the positive and negative (when needed) MS modes. Data were collected at full scan from 100 to 650 amu.

Entry compounds, carbomethoxyhydrazones **3**, were prepared from acetophenone (Ar = phenyl) or 2-acetylthiophene (Ar = 2-thienyl) **1**, following their condensation with methyl hydrazinecarboxylate **2** [30]. The preparation spiro(BID-pyrazoles) **7** and **8** involved the following procedure (Scheme 1).[1] Lithium diisopropylamide (LDA) (0.0788 mol) was prepared by the addition of 49 mL of 1.6 M *n*-butyllithium in hexanes (0.0788 mol) to a three-neck round-bottomed flask (e.g., 500 mL), equipped with a nitrogen inlet tube, a side-arm addition funnel (e.g., 125 mL), and a magnetic stir bar. The flask was cooled in an ice water bath and 8.02 g (0.0788 mol) of diisopropylamine, dissolved in 25–30 mL of THF, was added from the addition funnel at a fast drop wise rate during a 5 min period (0 °C, nitrogen). The solution was stirred for an additional 15–20 min, and then 0.0150 mol of the carbomethoxyhydrazone **3** dissolved in 50 mL of THF was added at a fast drop wise rate during 5–10 min. After 1 h, 3.39 g (0.0158 mol) of methyl 2-(aminosulfonyl)benzoate **5**, dissolved in 25–35 mL of THF was added, during 5 min, to the dilithiated intermediate **4**, and the solution was stirred and condensed for 1 h. Finally, 100 mL of 3 M hydrochloric acid was added quickly followed by 100 mL of solvent grade ether, then stirring the two-phase mixture for 5 min, followed by careful neutralization with solid sodium bicarbonate, and the two liquid phases or solid materials separated. If a solid appeared at this point, the biphasic mixture could be filtered. The aqueous layer was extracted with ether or THF (2 × 75 mL), and the organic fractions were combined, filtered, evaporated, and the solid organic materials were air dried. The twofold cyclization and acetylation required 1 mL of acetic anhydride and 6 mL of pyridine for each 1 g of dry

[1] CCDC for compound **7** (689212) and for compound **8** (689211) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge from the Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.



Scheme 1 Methyl *N*-acetyl spiro(benzoisothiazole–pyrazole)dioxide-*N*′-carboxylates

intermediate compounds [31]. Each gram of solid intermediate(s) is dissolved in 6 mL of pyridine followed by the drop wise addition of 1 mL of acetic anhydride. The solution is stirred at room temperature for 1 h. The addition of ca. 80 g of ice usually resulted in a precipitate, which was washed with water and recrystallized from ethanol or ethanol/benzene.

Methyl 2-acetyl-5′-phenyl-2*H*-spirobenzo[*d*]isothiazole-3,3′-pyrazole]-1,1-dioxide-2′(4′*H*)-carboxylate **7**; 67% yield mp 164–165 °C (ethanol/benzene); IR cm^{-1} 3036, 2979, 1727, 1709; ^{1}H NMR ($CDCl_3$) δ ppm 2.65 (s, 3H, CH_3), 3.69-4.05 (m, 5H), 7.06–7.91 (m, 7H, ArH); ^{13}C NMR ($CDCl_3$) δ ppm 23.7, 49.5, 53.6, 81.2, 121.2, 122.9, 127.6, 128.9, 129.2, 131.3, 132.5, 133.5, 135.3, 137.1, 147.7, 151.0, and 167.0. LC–MS, (M + H), 401.02; exact mass, 399.09, $[M + H]^+$, 399.42. Anal. calcd. for $C_{19}H_{17}N_3O_5S$: C, 57.13; H, 4.23; N, 10.52; found: C, 57.07; H, 4.15; N, 10.27.

Methyl 2-acetyl-5′-(2-thienyl)-2*H*-spiro[benzo[*d*]isothiazole-3,3′-pyrazole]-1,1-dioxide-2′(4′*H*)-carboxylate **8**, 67% yield mp 164–166 °C (ethanol/benzene); IR cm^{-1}, 3014, 2956, 1720, 1709; ^{1}H NMR ($CDCl_3$) δ ppm 2.65 (s, 3H, CH_3), 3.69–4.05 (m, 5H), 7.25–7.87(m, 7H, ArH); ^{13}C NMR ($CDCl_3$) δ ppm 24.1, 49.4, 53.5, 82.0, 121.7, 124.6, 128.6, 129.2, 130.2, 131.0, 132.2, 134.1, 136.5, 137.2, 149.1, 151.1 and 166.8. LC–MS, $[M + H]^+$, 406.01; exact mass, 405.44. Anal. calcd. for $C_{17}H_{15}N_3O_5S_2$: C, 50.36; H, 3.73; N, 10.36; found: C, 50.14; H, 3.63; N, 10.08.

Single Crystal X-ray Structure Determination

Yellow crystals of $C_{19}H_{17}N_3O_5S$ **7** and $C_{17}H_{15}N_3O_5S_2$ **8** were recrystallized from ethanol in order to give satisfactory crystals for X-ray determination. Crystal data for X-ray studies were collected at -120 °C on a Mercury CCD area detector coupled with a Rigaku AFC8 diffractometer with graphite monochromated Mo-K radiation. Data were collected in 0.50° oscillations in ω with 45 and 60 s exposures, respectively. A sweep of data was done using ω oscillations from $-40.0°$ to 90.0° at $\chi = 45.0°$ and $\varphi = 0.0°$; a second sweep was performed using ω oscillations from $-30.0°$ to 80.0° at $\chi = 45.0°$ and $\varphi = 90.0°$. The crystal-to-detector distances were 27.5904 mm for **7** and 27.8735 mm for **8**. Details of the data collection are reported in Tables 1 and 2. Data were collected, processed, and corrected for Lorentz polarization and for absorption using CrystalClear (Rigaku) [32, 33].

The non-hydrogen atoms were refined anisotropically. Ideal hydrogen atom coordinates were calculated and coordinates of the hydrogen atoms were allowed to ride on their respective carbon atoms. The temperature factors of all hydrogen atoms were varied isotropically with $U_{iso} = 1.2 \times U_{iso}$ of the carbon atom. Structure solution, refinement, and the calculation of derived results were performed using the SHELX-97 [34] package of computer programs. Neutral atom scattering factors were those of Cromer and

Table 1 Crystallographic data, $C_{19}H_{17}N_3O_5S$ **7** and $C_{17}H_{15}N_3O_5S_2$ **8**

CCDC deposit number	689212	689211
Color/shape	Yellow/parallelepiped	Yellow/needle
Crystal dimensions (mm)	0.48 × 0.24 × 0.05	0.24 × 0.10 × 0.07
Formula	$C_{19}H_{17}N_3O_5S$	$C_{17}H_{15}N_3O_5S_2$
Formula mass	399.42	405.44
T (°C)	−120(2)	−120(2)
Crystal system	Monoclinic	Orthorhombic
Space group	$P2_1/c$	$Pbca$
a (Å)	11.899(2)	16.045(3)
b (Å)	17.562(4)	10.746(2)
c (Å)	9.484(2)	20.389(4)
β (°)	111.03(3)	
V (Å^3)	1849.9(6)	3516(1)
Z	4	8
d_{calc} (g cm^{-3})	1.434	1.532
λ (Å)	0.71073	0.71073
μ (mm^{-1})	0.212	0.339
F(000)	832	1680
θ range (°)	3.61–25.15	3.23–25.14
Reflections collected	12415	4037
Miller indices	$-13 \le h \le 14$, $-21 \le k \le 21$, $-11 \le l \le 9$	$0 \le h \le 19$, $0 \le k \le 12$, $0 \le l \le 24$
Unique reflections	3295	3142
Unique reflections $I > 2\sigma(I)$	2207	2443
Max and min transmission	0.9895, 0.9049	0.9767, 0.9230
Data, restraints, parameters	3295, 0, 255	3142, 0, 246
Final R indices $I > 2\sigma(I)$	$R_1 = 0.0857$, $wR_2 = 0.2216$	$R_1 = 0.0841$, $wR_2 = 0.2179$
R indices all data	$R_1 = 0.1217$, $wR_2 = 0.2633$	$R_1 = 0.1054$, $wR_2 = 0.2448$
Goodness of fit on F^2	1.051	1.081
Largest diff peak and hole (e Å^{-3})	0.736, −0.610	0.571, −0.570

Table 2 Selected bond distances (Å), bond angles (°), and dihedral angles (°), $C_{19}H_{17}N_3O_5S$ **7** and $C_{17}H_{15}N_3O_5S_2$ **8**

	$C_{19}H_{17}N_3O_5S$	$C_{17}H_{15}N_3O_5S_2$
C1–C2	1.519(6)	1.510(7)
C2–C3	1.378(6)	1.375(7)
C3–S1	1.739(5)	1.744(5)
S1–N3	1.686(4)	1.686(4)
N3–C1	1.486(6)	1.496(6)
C1–N1	1.487(6)	1.474(6)
N1–N2	1.388(5)	1.401(5)
N2–C9	1.270(6)	1.295(6)
C9–C8	1.498(6)	1.496(7)
C8–C1	1.539(6)	1.538(6)
C9–C13	1.479(6)	1.436(7)
N1–C12	1.396(6)	1.370(6)
C12–O4	1.211(6)	1.208(6)
C12–O5	1.329(5)	1.329(6)
C13–C14		1.405(7)
C14–C15		1.433(8)
C15–C16		1.348(8)
C16–S2		1.706(5)
S2–C13		1.718(5)
C1–C2–C3	115.6(4)	116.1(4)
C2–C3–S1	111.6(4)	111.0(4)
C3–S1–N3	92.9(2)	93.5(2)
S1–N3–C1	115.7(3)	114.5(3)
N3–C1–C2	104.1(3)	104.5(4)
C1–C8–C9	103.2(4)	102.7(4)
C8–C9–N2	114.8(4)	114.9(4)
C9–N2–N1	107.4(4)	106.6(4)
N2–N1–C1	114.0(3)	113.7(4)
N1–C1–C8	99.9(3)	101.1(4)
C13–C14–C15		111.0(5)
C14–C15–C16		113.1(5)
C15–C16–S2		112.7(4)
C16–S2–C13		92.1(3)
S2–C13–C14		111.1(4)
C14–C13–C9–N2	171.3(5)	−174.6(5)
C13–C9–N2–N1	177.9(4)	−176.3(4)
N2–N1–C12–O4	−4.5(7)	−178.5(4)

Waber [35], and the real and imaginary anomalous dispersion corrections were those of Cromer [35].

Results and Discussion

When dilithiated acetophenone carbomethoxyhydrazone **4** (from **3**) was condensed with lithiated ester-sulfonamide **6**, we were readily able to isolate initial condensation product(s), *C*-acylated intermediates. Because of considerable difficulty in obtaining a single purified compound, the intermediates were not characterized. Further structural verification was initiated after cyclization of the intermediates to a single product **7** or **8**, which was easily purified by recrystallization from ethanol or ethanol/benzene. Acetic anhydride-pyridine at room temperature, an effective cyclization mixture, worked well [31], with the easy isolation of a product that contained an *N*-acetyl group (2-acetyl) resulting from an additional condensation

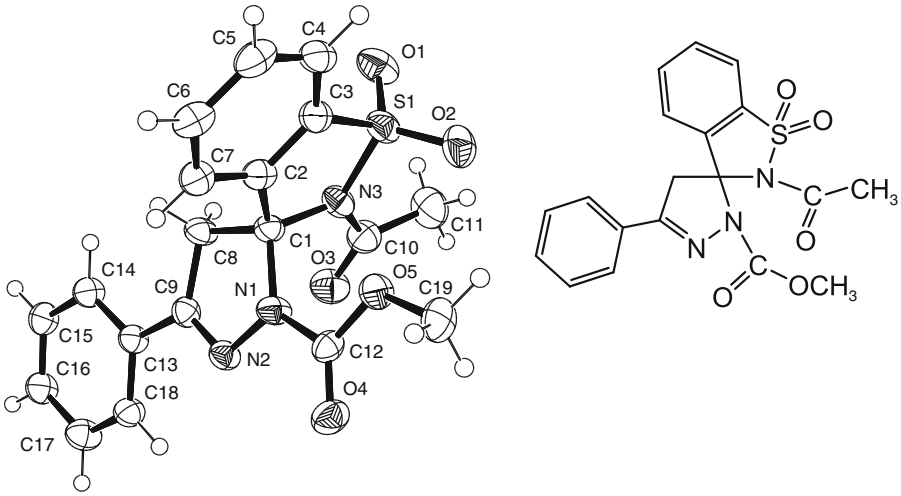


Fig. 1 ORTEP diagram (50% ellipsoids for non-hydrogen atoms) and illustration, $C_{19}H_{17}N_3O_5S$, **7** [36]

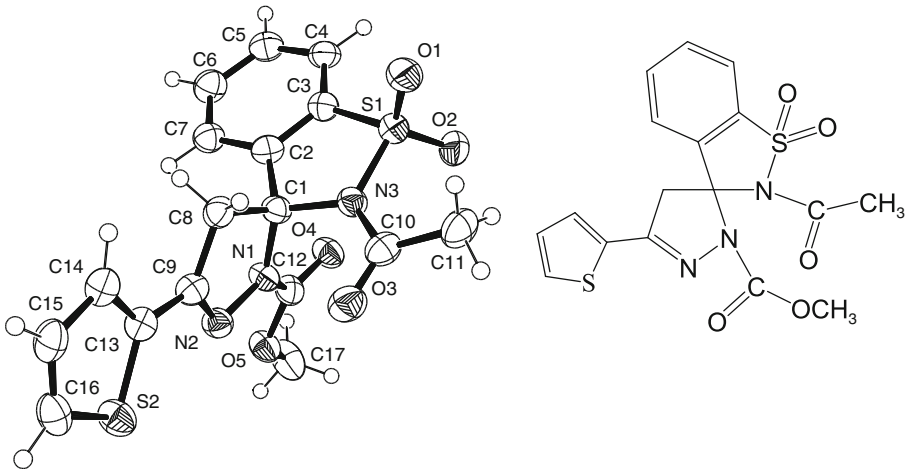


Fig. 2 ORTEP diagram (50% ellipsoids for non-hydrogen atoms) and illustration, $C_{17}H_{15}N_3O_5S_2$, **8** [36]

reaction. A second set of results involved the condensation of dilithiated 2-acetylthiophene carbomethoxyhydrazone **4** (from **3**) with **6** followed by separate cyclization of intermediates to **8** also in 67% yield.

Even though a straightforward mechanism could explain the unexpected *N*-acetyl spiro products **7** or **8**, due to their structural complexity, an X-ray crystal analysis of each compound was undertaken.

The molecular structures of $C_{19}H_{17}N_3O_5S$ **7** and $C_{17}H_{15}N_3O_5S_2$ **8** are shown in Figs. 1 and 2, and selected bond distances and angles are listed in Table 2. The bond lengths agree with the assignment of the double bond shown between C9 and N2 in both compounds. Because the unit cell of each compound is centrosymmetric, both enantiomorphic structures about the C1 chiral center are present.

The least squares best planes representing the fused rings are nearly coplanar with angles of 1.27° for **7** and 3.16° for **8**, respectively, between them. The two-five-member rings in each molecule are nearly perpendicular with angles of 89.67° for **7** and 88.04° for **8**, respectively. The rings connected by C9 and C13 are nearly coplanar with angles of 10.60° for **7**, and 9.91° for **8**, respectively, which allows for extended pi bonding between these rings and possibly with the atoms in the C12 carboxylate group.

In addition to the two conclusive ORTEP diagrams resulting from data for **7** and **8**, their structures were supported with absorption spectra and LC–MS.

Conclusions

X-ray analysis is important for the structure determination of methyl 2-acetyl-5′-(phenyl-2*H*-spiro[benzo[*d*]isothiazole-3,3′-pyrazole]-1,1-dioxide-2′(4′*H*)-carboxylate **7** and methyl 2-acetyl-5′-(2-thienyl)-2*H*-spiro[benzo[*d*]isothiazole-3,3′-pyrazole]-1,1-dioxide-2′(4′*H*)-carboxylate **8**, which resulted from the condensation–cyclization of dilithiated carbomethoxyhydrazones **4** prepared in excess LDA with lithiated methyl 2-(aminosulfonyl)benzoate **6**. The products resulted from condensation of an anionic nucleophile with the carbomethoxy carbon of lithiated ester-

sulfonamide **6**, followed by a selective cyclization process using acetic anhydride. These are not isolated results, and they are only an indication of the overall open endedness of the dilithiation of a large variety of appropriate $C(\alpha)$, *N*-carbomethoxyhydrazones **3** followed by condensation with this lithiated ester-sulfonamide **6**. The resulting intermediates have potential for cyclization to a host of heterocyclic products. As these investigations are initiated and developed some of the products may warrant X-ray crystallographic analysis.

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