bath. After approximately 1 h the temperature of the solution began to fall and the cooling bath was removed. The gas flow was continued for an additional 1 h, and the reaction mixture was then poured into 200 mL of water. The aqueous solution was extracted three times with 100 mL of ether. The ether layer was washed three times with 25 mL of water and dried (sodium sulfate). Evaporation of solvent and drying in vacuo over sodium hydroxide (to remove acetic acid) gave 11.2 g (55.8 mmol, 98%) of **5**, mp 50–51 °C (from ether/petroleum ether). The unrecrystallized product is of sufficient purity for the next stage. NMR ($CDCl_3$): δ 6.84 (d, J_{HF}^{meta} = 7.4 Hz, ArH-3), 6.63 (d, J_{HF}^{ortho} = 11.0 Hz, ArH-6), 4.61 (d, J_{HF} = 1.6 Hz, CH_2Cl), 3.89 (s, OCH_3), 3.87 (s, OCH_3).

Diethyl 2-Acetamido-2-(4,5-dimethoxy-2-fluorobenzyl)-malonate (4). Alkylation of 11 g (53.8 mmol) of **5** with the sodium salt of diethyl acetamidomalonate as described previously[5] gave, after recrystallization from aqueous ethanol, 17.1 g (44.4 mmol, 82.5%) of **4**: mp 140–142 °C (lit.[5] mp 136–138 °C); NMR (CD_3OD) δ 6.72 (d, J_{HF}^{ortho} = 11.1 Hz, ArH-3), 6.53 (d, J_{HF}^{meta} = 7.0 Hz, ArH-6), 4.89 (s, $ArCH_2$), 4.21 (m, OCH_2CH_3), 3.79 (s, OCH_3), 3.75 (s, OCH_3), 4.98 (s, $COCH_3$), 1.26 (t, J = 7.0 Hz, OCH_2CH_3).

Acknowledgment. D.C.F. was supported by NIH Grant No. F32AM07530.

Registry No. **2**, 102034-49-1; **3**, 71924-62-4; **4**, 102034-51-5; **5**, 91407-48-6; **6**, 398-62-9; 6-fluoronorepinephrine, 86820-21-5; 4-aminoveratrole, 6315-89-5; veratrole-4-diazonium tetrafluoroborate, 450-57-7; α,α-dichloromethyl ether, 4885-02-3; diethyl acetamidomalonate sodium salt, 1068-90-2.

A Convenient Preparation of 5-Alkyl-4-carbalkoxy-1,2,3-thiadiazoles

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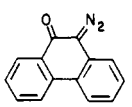
In connection with ongoing projects, preparation of 5-alkyl-4-carbalkoxy-1,2,3-thiadiazoles[1] was required. The reaction of the α-diazo β-ketoester **1** with 2,4-bis(4-methoxyphenyl)-1,3,2,4-dithiadiphosphetane 2,4-disulfide (**2**) or 2,4-bis(4-phenoxyphenyl-1,3,2,4-dithiadiphosphetane 2,4-disulfide (**3**) was envisioned as a direct entry to 1,2,3-thiadiazole **4** (eq 1—see Table I).

It has been described in the literature[2] that α-diazo thiocarbonyl compounds, such as **5**, could readily be cyclized to the 1,2,3-thiadiazole **6**. Substituted 1,2,3-thiadiazoles were also prepared from α-diazo ketones via their thioketone intermediates using Lawesson's reagent **2**.[3] However, it has been pointed out that only molecules possessing a rigid cis diazo ketone geometry could be converted to the 1,2,3-thiadiazoles. Thus, compound **7** was converted to **8**, whereas **9** did not yield the corresponding thiadiazole **10**.

5 **6**

As Cava and Levinson's suggestion was based on a single example and also the fact that **5** could readily be converted



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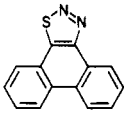


8

to the corresponding thiadiazole, it was decided to evaluate the reaction of diazo dicarbonyl compounds with both thionating reagents **2** and **3**. The results of this investigation are reported in this paper.

As shown in entries 1–4 (Table I), treatment of the α-diazo β-ketoester **1** (R = CH_3, Et; R′ = allyl) with 0.6 equiv of either **2** or **3** in refluxing benzene or THF, respectively, resulted in good to excellent yield of 1,2,3-thiadiazoles (76–94%).[4] Under these conditions both Lawesson's reagent[5] **2** and the more soluble 2,4-bis(4-phenoxyphenyl)-1,3,2,4-dithiadiphosphetane 2,4-disulfide[6] (**3**) reacted smoothly.[7] This procedure differs from Wolff's synthesis where ammonium hydrogen sulfide was used as thionating agent.[8,9]

The relative importance of steric hindrance[10] in the course of the reaction was examined in entries 5–7. The presence of a bulkier substituent adjacent to the ketone function required more forcing conditions. For example, substrate **1** (R = cyclopentyl; R′ = allyl) required a reaction time of 7 h under standard reaction conditions (entry 5). The bulkier diazo ketoester (R = *tert*-butyl; R′ = allyl) when refluxed for 20 h in THF gave the desired product in only 20% yield (entry 6).[11] However, when DME was used as a solvent, a 77% yield of the desired thiadiazole was obtained after 3.5 h (entry 7).

The modification of the ester side chain in **1** allows a quick entry to a variety of 5-alkyl-4-carbalkoxy-1,2,3-thiadiazoles (entries 8–11). Treatment of **1** (R = Et; R′ = CH_3, $CH_2CH_2Si(CH_3)_3$, benzyl, *tert*-butyl) with **3** under standard reaction conditions afforded the corresponding thiadiazole **4** in excellent yield.[12] These compounds can be readily transformed to the parent carboxylic acid **4** (R = Et; R′ = H) for further synthetic elaboration.[13]

In view of the above facile preparation of a variety of 5-alkyl-4-carbalkoxy-1,2,3-thiadiazoles, it was felt that conformationally unrestricted diazo ketones may also be

(1) The chemistry of 1,2,3-thiadiazoles has recently been reviewed; see: Thomas, E. W. In *Comprehensive Heterocyclic Chemistry*; Potts, K. T., Volume Ed.; Katritzky, A. R., Rees, C. W.; Series Eds.; Pergamon Press: London, 1984; Vol. 6, Part 4B, Chapter 4.24, p 447.

(2) (a) Regitz, M.; Liedhegener, A. *Justus Liebigs Ann. Chem.* **1967**, *710*, 118. (b) For a similar observation, see: Hinz, W.; Just, G. *Synth. Commun.* **1986**, 917.

(3) Levinson, M. I.; Cava, M. P. *Heterocycles* **1982**, *19*, 241.

(4) The more general synthesis of 1,2,3-thiadiazoles reported by Hurd and Mori where a hydrazone is treated with $SOCl_2$ is an interesting alternative to prepare 5-alkyl-4-carbalkoxy-1,2,3-thiadiazoles, although in some cases yields have been found to be irreproducible (Daris, J. P., Bristol-Myers, A.I. research, Candiac, private communication, 1985). See: (a) Hurd, C. D.; Mori R. I. *J. Am. Chem. Soc.* **1955**, *77*, 5359. (b) Shafiee, A. *J. Heterocycl. Chem.* **1976**, *13*, 301.

(5) A review on Lawesson's reagent has been published; see: Cava, M. P.; Levinson, M. I. *Tetrahedron* **1985**, *41*, 5061.

(6) For the application and the preparation of **3**, see: (a) Sauve, G.; Rao, V. S.; Lajoie, G.; Belleau, B. *Can. J. Chem.* **1985**, *63*, 3089. (b) Lajoie, G.; Lepine, F.; Maziak, L.; Belleau, B. *Tetrahedron Lett.* **1983**, *24*, 3815 and references cited therein.

(7) As expected the ester carbonyl was unreactive toward either of the thionating reagents.

(8) Wolff, L. *Justus Liebigs Ann. Chem.* **1902**, *325*, 129; **1904**, *333*, 1.

(9) For cases where Wolff's synthesis has been applied, see: (a) Wieland, H.; Bloch, S. *Chem. Ber.* **1906**, *39*, 1488. (b) Staudinger, H.; Becker, J.; Hinzel, H. *Chem. Ber.* **1916**, *49*, 1978, (c) Peet, N. P.; Sunder, S. *J. Heterocycl. Chem.* **1975**, *12*, 1191.

(10) The steric bulk effect could be felt either during the conversion of the C=O to C=S group or as steric compression between the groups around the C=C bond during the product-controlled formation of the final compound.

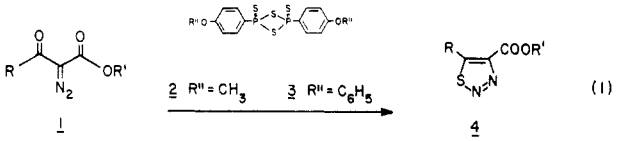
(11) The longer reaction time may give rise to consumption of **3** in side reactions, see: Pedersen, B. S.; Scheibye, S.; Clausen, K.; Lawesson, S. O. *Bull. Soc. Chim. Belg.* **1978**, *87*, 293.

(12) Detection of any of the presumably initially formed diazo thiocarbonyl compounds failed. For a similar observation, see ref 3.

(13) (a) See ref 4b and references cited therein. (b) Green, T. W. *Protective Groups in Organic Syntheses*; Wiley-Interscience: New York, 1981; Chapter 5, pp 152–192 and references cited therein.

0022-3263/86/1951-4075$01.50/0

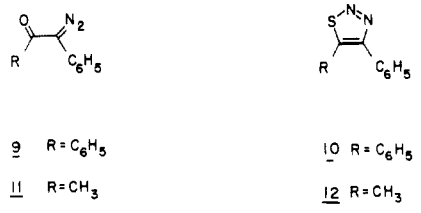
Table I. Preparation of 5-Alkyl-4-carbalkoxy-1,2,3-thiadiazoles[a]



entry	substrate R	substrate R'	thiophosphetane[b]	reactn condns	yield,[c] %
1	CH_3	allyl	2	refluxing benzene, 60 min	84
2	CH_3	allyl	3	refluxing THF, 45 min	76
3	Et	allyl	2	refluxing benzene, 60 min	92
4	Et	allyl	3	refluxing THF, 45 min	94
5	cyclopentyl	allyl	3	refluxing THF, 7 h	89
6	*tert*-butyl	allyl	3	refluxing THF, 20 h	20[d]
7	*tert*-butyl	allyl	3	refluxing DME, 3.5 h	77
8	Et	CH_3	3	refluxing THF, 45 min	95
9	Et	$CH_2CH_2Si(CH_3)_3$	3	refluxing THF, 45 min	88
10	Et	benzyl	3	refluxing THF, 45 min	84
11	Et	*tert*-butyl	3	refluxing THF, 45 min	89

[a] All reactions were carried out under an inert atmosphere with exclusion of moisture. [b] 0.6 equiv of Lawesson's reagent **2** or thiophosphetane **3** was used. [c] Yield of purified 1,2,3-thiadiazoles. [d] 80% of the starting material was recovered.

transformed to 1,2,3-thiadiazoles under the present experimental conditions. Azibenzil **9** when treated with Lawesson's reagent at room temperature (0.6 equiv, THF, 60 min) gave the thiadiazole **10** in 70% yield. In a similar manner the diazo ketone **11** gave the thiadiazole **12** in 43% yield.



9 R = C_6H_5 **10** R = C_6H_5

11 R = CH_3 **12** R = CH_3

These results appear to suggest that some conformationally flexible diazo carbonyl compounds may also be converted to the corresponding thiadiazoles through their α-diazo thiocarbonyl intermediates which must adopt the cis geometry in the transition state as suggested by Cava and Levinson.

In conclusion, it was shown that 5-alkyl-4-carbalkoxy-1,2,3-thiadiazoles are prepared in good yield from the corresponding α-diazo β-ketoesters by using either Lawesson's reagent **2** or the thiophosphetane **3**. Under similar reaction conditions, some conformationally unrestricted diazo ketones can also lead to the 1,2,3-thiadiazole ring system.

Experimental Section

Melting points were determined on a Büchi 510 melting point apparatus and are uncorrected. IR spectra were determined on a Perkin-Elmer Model 781 spectrophotometer. UV spectra were obtained on a Hewlett-Packard 8451 A UV spectrophotometer. NMR spectra were recorded in $CDCl_3$ on a Bruker AC-200 spectrometer. The frequency for proton was 200 MHz and for carbon was 50 MHz. Mass spectra were obtained on a Du Pont DP 102 GC/MS or a Hewlett-Packard 5985B GC/MS at 70 eV.

Microanalyses were performed by MICRO-TECH Laboratories Inc., Skokie, IL. Benzene was distilled from lithium aluminum hydride and tetrahydrofuran from sodium benzophenone ketyl. Lawesson's reagent was obtained from Aldrich and used without purification. Thiophosphetane **3** was prepared according to the literature procedure.[6b] All reactions were performed in oven-dried glassware under a positive pressure of argon or nitrogen. Crude products were purified by flash chromatography using 230–400 mesh silica gel (E. Merck).

Typical Procedure for the Thiadiazole Formation Using Lawesson's Reagent. A mixture of allyl 2-diazo-3-oxo-butanoate (228 mg, 1.36 mmol) and **2** (274 mg, 0.68 mmol) in 5.0 mL of dry benzene, under argon, was refluxed 60 min. The reaction mixture was concentrated and subjected to flash chromatography using 15:85 ethyl acetate/hexane as eluant. Treatment with decolorizing carbon afforded pure colorless 4-carballyloxy-5-methyl-1,2,3-thiadiazole (211 mg, 84% yield) as an oil. Analytical sample was prepared from bulb-to-bulb distillation: IR ($CHCl_3$) 3010, 1720, 1495, 1320, 1275, 1070 cm^{-1}; UV (cyclohexane) λ_{max} (ϵ) 254 (2219), 228 nm (3618); ^1H NMR δ 2.90 (s, 3 H), 4.93 (m, 2 H), 5.34 (m, 2 H), 6.04 (m, 1 H); ^{13}C NMR δ 10.88 (CH_3), 66.23 (CH_2), 119.34 (HC=), 131.36 (H_2C=), 149.89 (C-5), 160.1 (C-4), 160.4 (C=O); mass spectrum (70 eV), *m/e* (relative intensity) 184 (M^+, 12) 156 ($M^+ - N_2$, 3), 127 ($M^+ - OCH_2CH$=CH_2, 21). Anal. Calcd for $C_7H_8N_2O_2S$: C, 45.64; H, 4.38; N, 15.21; S, 17.41. Found: C, 45.27; H, 4.34; N, 15.30; S, 17.53.

Typical Procedure for the Thiadiazole Formation Using Thiophosphetane 3. A mixture of methyl 2-diazo-3-oxo-pentanoate (437 mg, 2.80 mmol) and reagent **3** (880 mg, 1.66 mmol) in 5.0 mL of dry THF, under argon, was refluxed 45 min. The reaction mixture was concentrated and subjected to flash chromatography using 15:85 ethyl acetate/hexane as eluant. Treatment with decolorizing carbon afforded pure colorless 4-carbomethoxy-5-ethyl-1,2,3-thiadiazole (457 mg, 95% yield) as an oil. Analytical sample was prepared from bulb-to-bulb distillation: IR ($CHCl_3$) 3010, 1725, 1495, 1330, 1310 cm^{-1}; UV (cyclohexane) λ_{max} (ϵ) 254 (2527), 224 nm (5616); ^1H NMR ($CDCl_3$) δ 1.40 (t, J = 7.46 Hz, 3 H), 3.37 (q, J = 7.46 Hz, 2 H), 4.02 (s, 3 H); ^{13}C NMR δ 15.88 (CH_3), 20.04 (CH_2), 52.59 (CH_3O), 148.87 (C-5), 161.22 (C-4), 168.25 (C=O); mass spectrum (70 eV), *m/e* (relative intensity) 172 (M^+, 7), 144 ($M^+ - N_2$, 36), 141 ($M^+ - OCH_3$, 7), 113 ($M^+ - N_2 - OCH_3$, 12). Anal. Calcd for $C_6H_8N_2O_2S$: C, 41.85; H, 4.68; N, 16.27; S, 18.62. Found: C, 41.36; H, 4.65; N, 16.23; S, 19.12.

5-Methyl-4-phenyl-1,2,3-thiadiazole (12). A mixture of diazo ketone **11** (600 mg, 3.75 mmol) and **2** (910 mg, 2.25 mmol) in 5.0 mL of dry benzene, under argon, was stirred at room temperature 60 min. The reaction mixture was concentrated and subjected to flash chromatography using 15:85 ethyl acetate/hexane as eluant. The known 5-methyl-4-phenyl-1,2,3-thiadiazole was obtained (284 mg, 43%) as a solid; mp 40–41 °C (lit.[14] mp 41.5 °C).

4,5-Diphenyl-1,2,3-thiadiazole (10). A mixture of diazo ketone **9** (376 mg, 1.69 mmol) and Lawesson's reagent (411 mg, 1.02 mmol) in 5.0 mL of dry benzene, under argon, was stirred at room temperature 60 min. The reaction mixture was concentrated and subjected to flash chromatography using 15:85 ethyl acetate/hexane as eluant. Recrystallization from methanol gave the known 4,5-diphenyl-1,2,3 thiadiazole (281 mg, 70%) as a white solid; mp 92–93 °C (lit.[4a] mp 92–93 °C).

(14) Schaumann, E.; Ehlers, J.; Forster, W. R.; Adiwidjaja, G. *Chem. Ber.* **1979**, 112, 1769.

Acknowledgment. I thank Drs. V. S. Rao, E. Ruediger, and H. Mastalerz for helpful discussions. The analytical research department (Syracuse, NY) is also acknowledged.

Registry No. 1 (R = CH_3, R′ = allyl), 91616-48-7; 1 (R = Et, R′ = allyl), 104034-79-9; 1 (R = cyclopentyl, R′ = allyl), 104034-80-2; 1 (R = *tert*-butyl, R′ = allyl), 104034-81-3; 1 (R = Et, R′ = CH_3), 104034-82-4; 1 (R = Et, R′ = $CH_2CH_2Si(CH_3)_3$), 104034-83-5; 1 (R = Et, R′ = benzyl), 86978-73-6; 1 (R = Et, R′ = *tert*-butyl), 104034-84-6; **2**, 19172-47-5; **3**, 88816-02-8; **4** (R = CH_3, R′ = allyl), 104034-71-1; **4** (R = Et, R′ = allyl), 104034-72-2; **4** (R = cyclopentyl, R′ = allyl), 104034-73-3; **4** (R = *tert*-butyl, R′ = allyl), 104034-74-4; **4** (R = Et, R′ = CH_3), 104034-75-5; **4** (R = Et, R′ = $(CH_2)_2Si(CH_3)_3$), 104034-76-6; **4** (R = Et, R′ = benzyl), 104034-77-7; **4** (R = Et, R′ = *tert*-butyl), 104034-78-8; **9**, 3469-17-8; **10**, 5393-99-7; **11**, 3893-35-4; **12**, 64273-28-5.

Supplementary Material Available: Full characterization data for all new compounds (2 pages). Ordering information is given on any current mashead page.

Mesyl Azide: A Superior Reagent for Diazo Transfer

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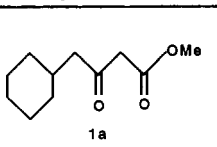
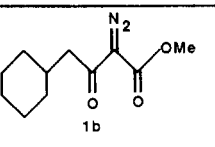
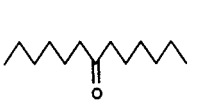
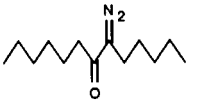
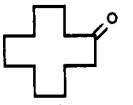
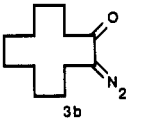
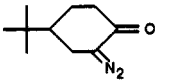
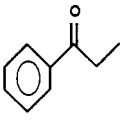
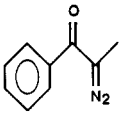
Received March 10, 1986

Base-catalyzed transfer of the diazo moiety to a methylene group adjacent to one or more electron-withdrawing groups is a well established and powerful synthetic tool.[2] The most commonly used reagent for diazo transfer has been *p*-toluenesulfonyl (tosyl) azide,[3] although isolated incidences of diazo transfer to β-dicarbonyl systems by other reagents have been reported.[4,5] Difficulties have been reported in the chromatographic separation of the desired product from excesss reagent and *p*-toluenesulfonamide following diazo transfer with tosyl azide.[6] Occasionally, there has been no choice but to use the resultant mixture in the subsequent step.[6b] We have found that methanesulfonyl (mesyl) azide is a generally superior reagent for diazo transfer.

The advantage of mesyl azide is that it is easily separated from the desired product upon washing the organic phase with 10% aqueous NaOH solution.[7] The use of *p*-carboxybenzenesulfonyl azide has been recommended[5a,6c] because of its solubility in base, but its high cost makes mesyl azide the better choice.

Mesyl azide is easily prepared in high yield from the inexpensive mesyl chloride and sodium azide in absolute MeOH, by the method of Boyer.[8] [Caution: *Although we have never had any trouble with mesyl azide, it is potentially explosive*!] Diazo transfer works well for both β-ketoesters and formyl ketones (Table I). The formyl ketones were not isolated; rather, mesyl azide was added directly to the pot containing the enolate resulting from formylation. This one-pot procedure is limited to symmetrical ketones, and ketones for which one enolate is preferred substantially over the other. Otherwise, a mixture of α-diazo ketones will result.

Table I

Entry	Substrate	Product	Yield[a]
1	 1a	 1b	94%
2	 2a	 2b	71%
3	 3a	 3b	66%[b]
4	 4a	 4b	68%
5	 5a	 5b	71%

[a] The yields are for pure chromatographed material. [b] The yield is based on recovered starting material (conversion was 83%).

Experimental Section

General Data. ^1H and ^{13}C NMR spectra were obtained on a Bruker WM-250 spectrometer as solutions in $CDCl_3$. Chemical shifts are reported in δ units downfield from the internal reference tetramethyl silane. The couplings (*J*) are reported in hertz (Hz). The infrared (IR) spectra were determined on a Nicolet 5DXB FTIR spectrometer as solutions in CCl_4 and are reported in reciprocal centimeters (cm^{-1}). Mass spectra (MS) were taken at 70 eV on a Du Pont 21-492B mass spectrometer and are reported as mass per unit charge (*m*/*z*), with intensities (as a percentage of the peak of greatest ion current having $m/z \geq 100$) in parentheses. Organic chemicals were purchased from Aldrich Chemical Co. Anhydrous ether was distilled from sodium metal

(1) Fellow of the Alfred P. Sloan Foundation 1982–1987.

(2) (a) Taber, D. F.; Schuchardt, J. L. *J. Am. Chem. Soc.* **1985**, *107*, 5289. (b) Chen, E. Y. *J. Org. Chem.* **1984**, *49*, 3245. (c) Crow, W. D.; Gosney, I.; Ormiston, R. A. *J. Chem. Soc., Chem. Commun.* **1983**, 643. (d) Adams, J. L.; Metcalf, B. W. *Tetrahedron Lett.* **1984**, *25*, 919. (e) Regitz, M.; Menz, F. *Chem. Ber.* **1968**, *101*, 2622.

(3) Regitz, M. *Angew. Chem., Int. Ed. Engl.* **1967**, *6*, 733.

(4) For previous uses of mesyl azide as a diazo-transfer reagent, see: (a) Lowe, G.; Ransay, M. V. J. *J. Chem. Soc., Perkin Trans. 1* **1973**, 479. (b) Stork, G.; Szajewski, R. P. *J. Am. Chem. Soc.* **1974**, *96*, 5787.

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