Minor lactone **10a** (23 mg, 2% from **3a**) as a white solid, treated immediately with HCl–MeOH to give the hydrochloride: spectroscopic data as for major lactone hydrochloride **10b**.

Debenzylation of 8b/9b Mixture. The above procedure was repeated using the **8b/9b** mixture (1.15 g), 10% palladium on charcoal (0.75 g), and methanol (130 mL) to give the following:

Major lactone **10b** (240 mg, 28% from **3b**) as a white solid: R_f 0.21; mp 128 °C; converted immediately by treatment with HCl–MeOH to its hydrochloride, obtained as white needles: mp 164–166 °C dec ($CHCl_3$–MeOH); $[\alpha]^{21}_D$ +49.7° (*c* 0.94, MeOH); IR (mull) 3060 w, 1770 cm^{-1}; ^1H NMR (D_2O) δ 1.28 (t, 1 H, *J* = 5.0 Hz), 1.63 (dd, 1 H, *J* = 5.0 and 8.3 Hz), 2.72 (m, 1 H), 4.10 (d, 1 H, *J* = 8.5 Hz), 4.33 (dd, 1 H, *J* = 8.5 and 4.8 Hz); ^{13}C NMR (D_2O) δ 17.0 (t), 23.3 (d), 38.8 (s), 71.4 (t), 174.9 (s). Anal. Calcd for $C_5H_8ClNO_2$: C, 40.15; H, 5.39; N, 9.36. Found: C, 40.26; H, 5.35; N, 9.27.

Minor ester **11b** (80 mg, 7% from *3b*) as an oil: spectroscopic data as for major ester **11a**.

Cyclodimerization of 10b Free Base. A solid sample of major lactone **10b** free base obtained by the above procedure was dissolved in chloroform to give an approximately 1.5 M solution, which was set aside at ambient temperature for 10 days. Evaporation of the solvent left a quantitative yield of **12b**, as a white solid: R_f 0.15 (10% MeOH in EtOAc); mp >250 °C; $[\alpha]^{24}_D$ +103.8° (*c* 0.27, MeOH); IR (mull) 3270 br, 3160 sh, 1645 cm^{-1}; ^1H NMR (CD_3OD) δ 1.43 (dd, 1 H, *J* = 6.1 and 9.8 Hz), 1.61 (dd, 1 H, *J* = 6.1 and 7.6 Hz), 1.75 (m, 1 H), 3.44 (d, 1 H, *J* = 11.3 and 9.5 Hz), 3.90 (dd, 1 H, *J* = 11.3 and 5.3 Hz); ^{13}C NMR (CD_3OD) δ 14.3 (t), 32.9 (d), 41.9 (s), 60.3 (t), 172.2 (s); MS (CI) *m/e* 227 (MH^+).

(1*S*,2*R*)-2-(Hydroxymethyl)-ACC, *trans*-4a. A mixture of ester **11a** (105 mg, 0.723 mmol) and 1 M sodium hydroxide solution (1.5 mL) was stirred at ambient temperature for 4 h, chilled in an ice bath, acidified to pH 2 with concentrated hydrochloric acid, and then lyophilized. The resulting white solids were dissolved in a minimum of water, and the solution was applied to a 1 cm × 10 cm column of Amberlite IRN-77 cation exchange resin (H^+ form), which was eluted with water until the eluent was neutral. The product was then eluted with 1 M ammonium hydroxide solution (60 mL). Evaporation to dryness left a white powder, which on double recrystallization from H_2O–EtOH at 0 °C gave *trans*-**4a** (71 mg, 75%) as fine white needles: fragment violently >195 °C, mp 232–234 °C dec; $[\alpha]^{24}_D$ −71.6° (*c* 1.04, H_2O); ^1H NMR (D_2O) δ 1.14 (dd, 1 H, *J* = 6.2 and 7.0 Hz), 1.46 (dd, 1 H, *J* = 1.2 and 10.0 Hz), 1.77–1.94 (m, 1 H), 3.71 (dd, 1 H, *J* = 6.8 and 12.0 Hz), 3.94 (dd, 1 H, *J* = 5.2 and 12.0 Hz); ^{13}C NMR (D_2O) δ 16.3 (t), 25.7 (d), 40.6 (s), 59.8 (t), 176.7 (s). Anal. Calcd for $C_5H_9NO_3$: C, 45.80; H, 6.92; N, 10.68. Found: C, 44.91; H, 7.12; N, 10.41.

(1*S*,2*S*)-2-(Hydroxymethyl)-ACC, *cis*-4b. The above procedure was repeated using lactone hydrochloride **10b** (70 mg, 0.468 mmol) and 1 M sodium hydroxide solution (1.35 mL), and the crude product obtained as a fine white powder, recrystallized from H_2O–EtOH at −20 °C to give *cis*-**4b** (42 mg, 68%) as a clump of plates: sinter >150 °C to slender needles, mp 198–200 °C dec; $[\alpha]^{24}_D$ +23.3° (*c* 0.95, H_2O); ^1H NMR (D_2O) δ 1.38 (dd, 1 H, *J* = 10.2 and 6.5 Hz), 1.42 (t, 1 H, *J* = 6.5 Hz), 1.72–1.82 (m, 1 H), 3.75 (dd, 1 H, *J* = 11.5 and 7.2 Hz), 3.81 (dd, 1 H, *J* = 11.5 and 6.7 Hz); ^{13}C NMR (D_2O) δ 16.3 (t), 27.5 (d), 40.1 (s), 59.8 (t), 174.2 (s). Anal. Calcd for $C_5H_9NO_3 \cdot {}^1/_2H_2O$: C, 42.85; H, 7.19; N, 10.00. Found: C, 42.54; H, 6.99; N, 10.27.

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Registry No. 1, 101986-32-7; (±)-**2a**, 13403-37-7; (±)-**2b**, 82584-73-4; (*R*)-**2b**, 51594-57-1; (*S*)-**2b**, 96479-96-8; (±)-**2c**, 125876-09-7; (±)-**2d**, 118712-54-2; (±)-**2e**, 82337-76-6; (1*S*,2*R*)-**3**, 119066-45-4; (1*R*,2*R*)-**3**, 119066-46-5; (1*S*,2*S*)-**3**, 118970-47-1; (1*R*,2*S*)-**3**, 119066-44-3; *cis*-**46**, 125876-14-4; *trans*-**4a**, 125876-15-5; **5d**, 125830-44-6; **6**, 125830-45-7; **8a**, 119068-14-3; **8b**, 119068-17-6; **9a**, 114498-09-8; **9b**, 119066-47-6; **10a** (free base), 125876-11-1; **10a**·HCl, 119068-16-5; **10b** (free base), 125876-12-2; **10b**·HCl, 119068-15-4; **11a**, 114498-10-1; **11b**, 119066-43-2; **12a**, 125830-47-9; **12b**, 125876-13-3; **13a**, 125830-46-8; **13b**, 125876-10-0.

2-(((*p*-Nitrophenyl)sulfonyl)oxy)-3-keto Esters: Versatile Intermediates for the Preparation of 1,2,3-Tricarbonyl Compounds†

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The excellent leaving ability of the nosylate group and the high, differentiated functional group density in 2-(((*p*-nitrophenyl)sulfonyl)oxy)-3-keto esters, **1**, suggested that they might serve as versatile precursors for the synthesis of other 1,2,3-trifunctionalized compounds. Reaction of 2-(nosyloxy)-3-keto esters with triethylamine gives 1,2,3-tricarbonyl compounds in high yields. The tricarbonyl compound can be reacted, without isolation, with nucleophiles to give heterocyclic products in excellent yields.

Interest in 1,2,3-tricarbonyl compounds has risen dramatically in the last few years. The occurrence of this functionality in the powerful immunosuppressent FK-506[1] and related antibiotics[2] has led to several strategies for its incorporation into target structures.[3] In addition, Wasserman has elegantly demonstrated the valuable reactivity patterns of vicinal tricarbonyl compounds in the synthesis of a variety of heterocyclic systems.[4]

The best current methods for the introduction of the 1,2,3-tricarbonyl group include the singlet oxygen cleavage or ozonolysis of 2-enamino-3-keto esters,[4f] the ozonolysis of 2-iodoniumyl-3-keto esters,[5] the singlet oxygen cleavage or ozonolysis of 2-phosphorous ylide derivatives of 3-keto esters,[3] and the hydrolysis of 2-oximino-3-keto esters.[4e]

We have recently found that 2-(((*p*-nitrophenyl)-

† This paper is dedicated to Professor Ralph L. Dannley of Case Western Reserve University on the occasion of his 75th birthday.

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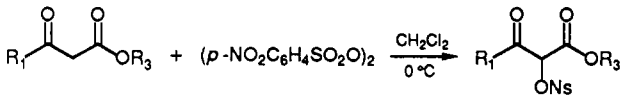
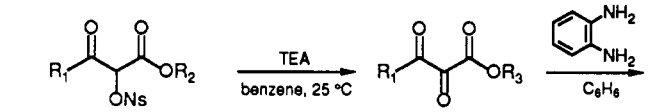
sulfonyl)oxy)-3-keto esters, 1, are readily prepared by the reaction of 3-keto esters with p-nitrobenzenesulfonyl peroxide[6] (eq 1). The excellent leaving ability of the nosylate group and the high, differentiated functional group density in 1 suggested that they might serve as versatile precursors for the synthesis of other 1,2,3-trifunctionalized compounds. We report that 2-(nosyloxy)-3-keto esters are easily converted to 1,2,3-tricarbonyl compounds in high yields.



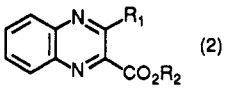
(1)

Treatment of 2-(nosyloxy)-3-keto esters **1a–d** with triethylamine in benzene at room temperature led to the rapid disappearance of the starting material. Attempts to isolate the tricarbonyl products **2a–d** were unsuccessful, as only low yields could be separated by column chromatography. This was not completely unexpected since 1,2,3-tricarbonyl compounds are often difficult to isolate in a pure state.[5,7]

The tricarbonyl product could be trapped in situ as the quinoxaline derivative **3a–d** by the addition of *o*-phenylenediamine to the crude product mixture and refluxing for 2 h (eq 2). The generally high yields shown in eq 2, which are isolated yields of pure product, indicate that nosylates **1a–d** give tricarbonyl compounds **2a–d** and hence the quinoxalines **3a–d** very efficiently.



1a: $R_1 = CH_3$; $R_2 = Et$
b: $R_1 = CH_3$; $R_2 = Me$
c: $R_1 = CH(CH_3)_2$; $R_2 = Et$
d: $R_1 = Ph$; $R_2 = Et$

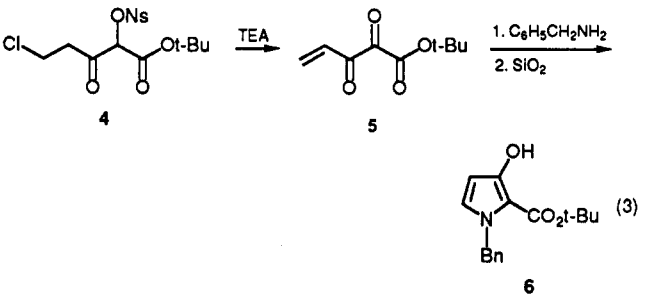


(2)

3a: 74%
b: 94%
c: 84%
d: 92%

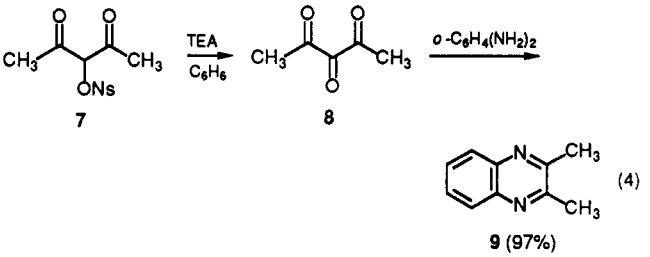
The conversion of keto ester nosylates 1 to the tricarbonyl products 2 probably occurs by base-promoted, reductive elimination of *p*-nitrobenzenesulfinate from the anion of 1, a process which has been previously observed in 2-sulfonyloxy esters.[8] Simply treating **1a** with *o*-phenylenediamine at room temperature fails to give **3a**, presumably because the pK_a of *o*-phenylenediamine is too low to remove the proton at C-2 to a significant extent. Other processes between **1a** and *o*-phenylenediamine which might give **3a**, but which do not involve tricarbonyl **2a**, are also ruled out.

Chloro keto ester nosylate **4**, prepared from *tert*-butyl 5-chloro-3-oxopentanoate,[9] underwent a double elimination in the presence of triethylamine to the vinyl tricarbonyl **5** (eq 3).[4f] This product could be crystallized from the reaction mixture in low yields; however, addition of benzylamine (1 equiv) to the crude product mixture followed by silica gel gave the pyrrole derivative **6** (58%), which is known to be produced by the reaction of benzylamine with **5**.[4f] Alternatively, treatment of **4** with benzylamine (3 equiv) gave **6** directly (68%). Thus benzylamine is sufficiently basic to promote the conversion of **4** to **5**, in addition to trapping **5** as it is produced. Similar behavior is expected for other trapping agents with pK_a's > 9.



(3)

Analogous to the results for keto ester nosylates **1a–d** and **4**, β-diketonosylate **7** underwent smooth elimination to triketone **8**, which was trapped as the quinoxaline **9** (97%) (eq 4).



(4)

The method reported here is an attractive alternative to other methods for the preparation of 1,2,3-tricarbonyl compounds since it is a simple, two-step process from readily available 3-keto esters. While it is difficult to isolate the tricarbonyl product from the reaction mixture, the tricarbonyl compound can be reacted without isolation with nucleophiles to give the derived products in generally excellent yields. For instance, the conversion of *tert*-butyl 5-chloro-3-oxopentanoate to hydroxypyrrole **6** can be accomplished in two steps in 48% yield. For comparison purposes, the same starting material has been converted to **6** by ozonolysis of an enamine derivative, dehydrochlorination, and condensation with benzylamine in 27% overall yield after four steps. Alternatively **6** can be prepared in 38% yield via a phosphorous ylid derivative in four steps.[4f]

Experimental Section

Melting points were obtained on a Mel Temp apparatus and are uncorrected. Infrared spectra were taken on a Perkin-Elmer 283 spectrophotometer. Proton NMR spectra were obtained on a Varian XL-200 instrument. Chemical shifts are reported for chloroform-*d* solution in ppm relative to Me_4Si. Elemental analyses were performed by Desert Analytics, Tuscon, AZ. Thin-layer chromatography was performed on silica gel 60 F_{254} plates from EM Reagents and visualized by UV irradiation and/or iodine. Flash column chromatography was performed using silica gel 60 (230–400 mesh).[10] The 2-(nosyloxy)-3-keto ester starting materials **1a-d** and **4** were prepared as described in ref 6.

General Procedure for the Preparation of Quinoxalines. To a stirred solution of a 2-(nosyloxy)-3-keto ester (1 mmol)[6] in benzene (80 mL) was added triethylamine (0.5 mL), and the mixture was stirred at room temperature for 1 h. *o*-Phenylene-

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diamine (2 mmol) and *p*-TsOH (50 mg) was added to the resulting pale yellow solution which was then refluxed for 2 h and concentrated on a rotary evaporator. The residue was dissolved in ethyl acetate (80 mL), washed with 1 N HCl (50 mL) and saturated $NaHCO_3$ (50 mL), passed through a short pad of $MgSO_4$ and silica gel 60, and concentrated to provide a pale yellow oil. Purification by flash column chromatography (hexane–ethyl acetate, 90:10) gave the quinoxaline as a white solid.

Quinoxaline 3a was prepared from **1a** (1 mmol) by the above general procedure in 74% yield: mp 65–66 °C; NMR ($CDCl_3$) δ 1.50 (t, 3 H, OCH_2CH_3), 2.96 (s, 3 H, CH_3), 4.57 (q, 2 H, OCH_2CH_3), 7.75–8.30 (m, 4 H, aromatic CH); IR (CH_2Cl_2) 3040, 2970, 1720, 1550 cm^{-1}. Anal. Calcd for $C_{12}H_{12}N_2O_2$: C, 66.67; H, 5.56; N, 12.96. Found: C, 66.29; N, 5.55; N, 12.74.

Quinoxaline 3b was prepared from **1b** by the same general procedure in 94% yield: mp 79–80 °C; NMR ($CDCl_3$) δ 2.87 (s, 3 H, $C(=N)CH_3$), 3.98 (s, 3 H, OCH_3), 7.60–8.1 (m, 5 H, aromatic CH). Anal. Calcd for $C_{11}H_{10}N_2O_2$: C, 65.34; H, 4.95; N, 13.86. Found: C, 64.83; H, 4.86; N, 13.59.

Quinoxaline 3c was prepared from **1c** by the same general procedure in 84% yield: mp 178–182 °C dec; NMR ($CDCl_3$) δ 1.43 (d, 6 H, J = 6.8 Hz, $CH(CH_3)_2$), 1.49 (t, 3 H, OCH_2CH_3), 3.70 (sept, 1 H, J = 6.8 Hz, $CH(CH_3)_2$), 4.57 (q, 2 H, OCH_2CH_3), 7.77 (m, 2 H, aromatic CH), 8.13 (m, 2 H, aromatic CH); IR (CH_2Cl_2) 3100, 2980, 1725, 1525 cm^{-1}. Anal. Calcd for $C_{14}N_{16}N_2O_2$: C, 68.85; H, 6.56; N, 11.48. Found 69.11; H, 6.74; N, 11.35.

Quinoxaline 3d was prepared from **1d** by the same general procedure in 92% yield: mp 51–53 °C; NMR ($CDCl_3$) δ 1.17 (t, 3 H, OCH_2CH_3), 4.33 (q, 2 H, OCH_2CH_3), 7.50 (m, 3 H, aromatic CH), 7.82 (m, 4 H, aromatic CH), 8.20 (m, 2 H, aromatic CH); IR (CH_2Cl_2) 3050, 2970, 1730, 1540 cm^{-1}. Anal. Calcd for $C_{17}H_{14}N_2O_2$: C, 73.38; H, 5.04; N, 10.07. Found: C, 73.67; H, 4.89; N, 9.82.

Quinoxaline 9 was prepared from 2-(nosyloxy) β-diketone **7** by the same general procedure in 97% yield: mp 83–86 °C; NMR ($CDCl_3$) δ 2.82 (s, 3 H, $C(=N)CH_3$), 2.94 (s, 3 H, $C(=O)CH_3$), 7.76 (m, 2 H, aromatic CH), 8.05 (m, 2 H, aromatic CH); IR (CH_2Cl_2) 1690, 1540 cm^{-1}. Anal. Calcd for $C_{11}H_{10}N_2O$: C, 70.97; H, 5.38; N, 15.05. Found: C, 70.73; H, 5.48; N, 15.01.

***tert*-Butyl 5-chloro-3-oxopentanoate** was prepared by a slight modification of the literature procedure.[9] To a stirred and cooled (0 °C) solution of *N*-isopropylcyclohexylamine (3.3 mL, 20 mmol) in dry THF (10 mL) was added 2.5 M of *n*-BuLi in hexane (8 mL, 20 mmol). After being stirred for 10 min, the mixture was cooled to −78 °C. *tert*-Butyl acetate (3.0 mL, 22 mmol) was added, and the mixture was stirred at −78 °C for 30 min. A solution of ethyl 3-chloropropionate (1.37 g, 10 mmol) in THF (20 mL) was cooled to −78 °C, and the solution of the lithium enolate of *tert*-butyl acetate prepared above was added via syringe. After stirring at −78 °C for 5 min, the reaction was quenched with acetic acid (10 mL) and poured into a mixture of ethyl ether–water (150:50 mL). The organic phase was separated, washed with 20% aqueous potassium carbonate (3 × 50 mL) and brine (100 mL), dried ($MgSO_4$), and concentrated to provide *tert*-butyl 5-chloro-3-oxopentanoate (100%): NMR ($CDCl_3$) δ 1.48 (s, 9 H, $C(CH_3)_3$), 3.04 (t, 2 H, $ClCH_2CH_2CO$), 3.40 ($COCH_2CO$), 3.75 (t, 2 H, $ClCH_2CH_2CO$). The crude product was used for the next reaction without further purification.

***tert*-Butyl 5-Chloro-2-(((*p*-nitrophenyl)sulfonyl)oxy)-3-oxopentanoate (4).** To a cooled (0 °C) solution of *tert*-butyl 5-chloro-3-oxopentanoate (2.07 g, 10 mmol) in ethyl acetate (200 mL) was added *p*-nitrobenzenesulfonyl peroxide (4.04 g, 10 mmol)[11] and powdered anhydrous potassium carbonate (1.0 g). The reaction was stirred for 3 h at 0 °C and was allowed to warm to room temperature overnight. The resulting suspension was washed with 1 N HCl (3 × 100 mL) and brine (100 mL), passed through a short pad of $MgSO_4$ and silica gel 60, and concentrated to give a white solid. Purification by flash chromatography (hexane–ethyl acetate, 80:20) provided **4** as a white solid (2.90 g, 71%) with mp 88–89 °C: NMR ($CDCl_3$) δ 1.44 (2 s, 9 H, $C(CH_3)_3$), 3.15 (2 t, 2 H, $COCH_2CH_2Cl$), 3.75 (t, 2 H, $COCH_2CH_2Cl$), 5.33 (s, 0.6 H, CHONs). The ^1H NMR spectrum was consistent with a 5:3 ratio of keto:enol tautomers. Anal. Calcd for $C_{15}H_{18}NO_8SCl$: C, 44.17; H, 4.42; N, 3.44. Found: C, 43.95; H, 4.28; N, 3.54.

***tert*-Butyl 1-Benzyl-3-hydroxypyrrole-2-carboxylate (6).** To a stirred, cooled (0 °C) solution of *tert*-butyl 5-chloro-2-(((*p*-nitrophenyl)sulfonyl)oxy)-3-oxopentanoate, **4** (230 mg, 0.57 mmol), in dichloromethane (50 mL) was added triethylamine (0.3 mL) slowly, followed by water (0.1 mL). After the mixture was stirred for 1 h at 0 °C, benzylamine (70 μL, 0.6 mmol) was added, and the mixture was stirred at room temperature for 1 h. Silica gel (3.0 g) was added, and stirring was continued overnight. The resulting suspension was filtered on short pad of silica gel 60, and the filtrate was concentrated. Purification by preparative silica gel TLC (hexane–ethyl acetate, 95:5) provided **6** as colorless oil (90 mg, 58%) which slowly solidified. The spectral data of **6** matched the literature values.[12]

Alternatively 3 equiv of benzylamine were added to a solution of **4**, followed by water (0.1 mL), and the mixture stirred for 1 h at room temperature. Addition of silica gel as above and normal workup gave **6** (68%).

Acknowledgment. This work was made possible by a grant from the National Science Foundation (87-09853).

Registry No. **1a**, 124716-79-6; **1b**, 124716-78-5; **1c**, 124716-80-9; **1d**, 124716-83-2; **3a**, 3885-38-9; **3b**, 61522-54-1; **3c**, 125879-67-6; **3d**, 3885-46-9; **4**, 125879-66-5; **6**, 65171-95-1; **7**, 124716-87-6; **9**, 2379-55-7; $H_2NC_6H_4$-*o*-NH_2, 95-54-5; $(H_3C)_3COAc$, 540-88-5; $ClCH_2CH_2C(O)OEt$, 623-71-2; $ClCH_2CH_2C(O)CH_2C(O)OBu$-*t*, 88023-66-9; *p*-$O_2NC_6H_4SO_2$-O-O-$SO_2C_6H_4$-*p*-$NO_2$, 6209-72-9; $PhCH_2NH_2$, 100-46-9.

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