Cyclobutenone Ethylenedithioacetals and Their Ready Electrocyclic Ring Opening

Wilko Regenhardt,* Ernst Schaumann,* Harold W. Moore*b

*a Institut für Organische Chemie, Technische Universität Clausthal, Leibnizstraße 6, 38678 Clausthal-Zellerfeld, Germany
Fax +49(5323)722858; E-mail: ernst.schaumann@tu-clausthal.de

b Department of Chemistry, University of California-Irvine, Irvine, CA 92697–2025, USA
Fax +1(949)8242210; E-mail: halmoore@uci.edu

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Abstract: Reported here is a general regiospecific synthesis of cyclobutenedione monoethylendithioacetals which readily undergo ring opening after addition of an organolithium reagent. The generated acyclic enols either tautomerize to the corresponding carbonyl compounds or can be trapped as silylenol ethers, which serve as electron rich dienes in Diels–Alder additions with tetracyanoethylene or maleic anhydride.

Key words: cyclobutenones, dithioacetals, transthioacetalization, electrocyclic ring opening, Diels–Alder reaction

Cyclobutenone derivatives have been efficiently used for the synthesis of highly substituted p-quinones and related annulated compounds over the last 15 years.1–3 The thermal ring expansion is presumed to proceed via ring opening of the cyclobutenone to a vinyl ketene intermediate which then undergoes electrocyclic ring closure to form the six-membered ring. Starting materials of particular note are cyclobutenedione monoketals. Such compounds having predictable regiochemistry are readily prepared and serve as useful precursors to asymmetrically substituted p-quinones.4,5

Our interest in organosulfur derivatives of cyclobutenones6 led us to investigate the chemistry of cyclobutenedione dithioacetals. In particular, we were interested in 1,3-dithiolane derivatives because of their potential utility for the generation of cyclobutenediones via a base induced [3 + 2] cycloreversion reaction.7

Reported herein is an efficient preparation of cyclobutenedione monodithioacetals involving the transthioacetalization of the corresponding dialkyl acetals. Firouzabadi and Iranpoor8 have developed a method for a selective transthioacetalization of open chain acetals in the presence of cyclic acetals by the use of catalytic amounts of ZrCl4. This methodology could be extended to the transthioacetalization of cyclobutenedione monoketals. Thus, the readily available dimethyl acetals 1 were converted to the corresponding 1,3-dithiolanes 2 in the presence of 1,2-ethanediol (1.05 equivalents) and ZrCl4 (15 mol%) in very good yields and with complete control of chemoselectivity (Scheme 1).

Table 1 Transthioacetalization with ZrCl4

| Product | R1 | R2 | Yield (%) | Mp (°C) |
|---------|----|----|-----------|---------|
| 2a      | Bu | Me | 83        | oil     |
| 2b      | t-Bu | Me | 85        | 89–90   |
| 2c      | Ph | Me | 88        | 104–105 |
| 2d      | t-Bu | vinyl | 79 | oil       |
| 2e      | Bu | OMe | 86 | oil     |
| 2f      | t-Bu | OMe | 84 | 100–101 |
| 2g      | Ph | OMe | 94 | 148–150 |
| 2h      | C=C-Bu | OMe | 80 | oil     |

The symmetrically substituted cyclobutenedione monooethylendithioacetal 2i was prepared by a BF3·OEt2 catalyzed thioacetalization of 3,4-dimethylcyclobutenedione (3) (Scheme 2). The degree of the accompanying bisthioacetalation was reduced by a very slow addition of a CH2Cl2 solution of ethanediol and BF3·OEt2 to a solution of the dione in CH2Cl2 at 0 °C. However, under these conditions, we obtained 8% of the bisthioacetal 4 and 73% of the desired monoethylendithioacetal 2i.
The cyclobutenedione monoethylendithioacetals 2 were treated with an organolithium reagent (R\textsuperscript{3}Li) in THF at −78°C followed by an aqueous work up to furnish the ring opening products 6 (Scheme 3, Table 2). Thus, in contrast to the stable 4-hydroxy-cyclobutenedione dialkyl acetals,\textsuperscript{3,4} the dithiolane derivatives readily undergo ring opening and subsequent tautomerization of the primary enols to the ketones 6 under the reaction conditions.

![Scheme 3](image)

**Scheme 3**

Quenching of the 1,2-adducts 5 with chlorotrimethylsilane led to the vinyl ketenethioacetals and subsequent tautomerization of the primary enols to the dithiolane derivatives readily undergo ring opening.

**Table 2** 1,2-Addition and Subsequent Electroyclic Ring Opening to Ketones 6 and Silylenol Ethers

| Product | R\textsuperscript{1} | R\textsuperscript{2} | R\textsuperscript{3} | Method\textsuperscript{a} | Yield (%) |
|---------|-----------------|-----------------|-----------------|-----------------|-----------|
| 6a      | Me              | Me              | Ph              | A               | 73        |
| 6b      | Bu              | OMe             | Ph              | A               | 71        |
| 6c      | tert-Bu         | OMe             | Ph              | A               | 73        |
| 7a      | tert-Bu         | OMe             | Bu              | B               | 81        |
| 7b      | tert-Bu         | OMe             | vinyl           | B               | 65        |
| 7c      | tert-Bu         | OMe             | Ph              | B               | 79        |
| 7d      | Bu              | OMe             | Ph              | B               | 77        |
| 7e      | tert-Bu         | OMe             | C=C-Bu          | C               | 90        |

\textsuperscript{a} See experimental.

Dienes 7 warrant further study as multifunctional synthetic building blocks; it is noted that they are thermally stable and do not cyclize even in refluxing p-xylene solution. Even more flexibility can be expected for sterically less hindered derivatives with R\textsuperscript{3}=H, e.g., diene 11 (Scheme 4).

The synthesis of 11 started with the reduction of cyclobutenedione 2i with DIBALH in THF at 0°C to give alcohol 8 in 88% yield. Subsequent ring opening of the secondary alcohol to the acyclic aldehyde 9 was completed in 4 hours at 50°C. At room temperature, this ring opening is comparatively slow. Thus, isolation and subsequent silylation of the alcohol 8 by tert-butylimidylsilyl triflate (TBSOTf), triethylamine and 4-DMAP in CH\textsubscript{2}Cl\textsubscript{2} at 0°C were possible and allowed clean conversion to the silyl ether 10 in 92% yield. The silyl enol ether 11 was obtained in 99% yield by heating a solution of 10 in CHCl\textsubscript{3} at 50°C for 4 hours.

To illustrate the utility of diene 11 in Diels–Alder cycloaddition reactions, it was treated with tetracyanoethylene (TCNE) in CHCl\textsubscript{3} at room temperature to give the desired adduct 12 within 30 minutes in 97% yield. The cycloaddition with maleic anhydride required the higher reaction temperature of refluxing toluene and led to the cycloadduct 13 in 52% yield.

**Scheme 5** a) Reagents and conditions: TCNE, CHCl\textsubscript{3}, r.t., 15 min; b) maleic anhydride, toluene, reflux, 24 h

The scope of these [4 + 2] cycloadditions is so far limited to reactive dienophiles.

NMR spectra were recorded on a Bruker ARX-400 or G.E. Omega 500 spectrometer using CDCl\textsubscript{3} or TMS as internal standard. IR spectra were recorded on a Perkin–Elmer 1600 FT-IR spectrometer. MS were recorded on a VG Analytic 7070E instrument. THF was dried by passing through two 4 x 36 in.\textsuperscript{2} columns of anhyd neutral A-2 alumina. CH\textsubscript{2}Cl\textsubscript{2} and toluene were distilled from CaH\textsubscript{2}. All reactions were followed by TLC using Merck precoated plates of silica gel 60 F\textsubscript{254}. Merck silica gel 60 (mesh 230–400) was used in
flask chromatography. Solvents for chromatography were distilled prior to use.

**Cyclobutenedione Monoethylendithioacetals 2; General Procedure**

A solution of cyclobutenedione monodimethylacetal 1 (1 mmol) and 1,2-ethanedithiol (87 μL, 1.05 mmol) in dry CH₂Cl₂ (5 mL) was stirred at 0°C when ZrCl₄ (35 mg, 0.15 mmol) was added. After 2 h at 0°C, the reaction mixture was quenched with NaOH (10%, 5 mL). The aqueous layer was separated and extracted with CH₂Cl₂ (2 × 5 mL). The combined organic layers were washed with H₂O (10 mL), then with brine (10 mL), and dried. Removal of the solvent in vacuo followed by flash chromatography (hexanes–EtOAc) on silica gel provided the desired product.

**Thioacetalization of 3,4-Dimethylcyclobutenedione 3**

A solution of 1,2-ethanedithiol (0.42 mL, 5 mmol) and BF₃·OEt₂ (0.65 mL, 5 mmol) was added dropwise over 3 h to a solution of 3,4-dimethylcyclobutenedione 3 (550 mg, 5 mmol) in dry CH₂Cl₂ (5 mL). After complete addition the resulting solution was stirred at r.t. for 1 h and then neutralized with sat. NaHCO₃ (30 mL). The aqueous layer was separated and extracted with CH₂Cl₂ (2 × 20 mL). The combined organic layers were washed with brine (30 mL) and dried. Removal of solvent in vacuo followed by flash chromatography (hexanes–EtOAc, 20:1) provided products 2i and 4.

| Product | IR v (cm⁻¹) | ¹H NMR (CDCl₃; 500 MHz) δ (ppm), J (Hz) | ¹³C NMR (CDCl₃; 100 MHz) δ (ppm) | MS m/z |
|---------|------------|----------------------------------------|---------------------------------|--------|
| 2a      | 2958, 2929, 1765, 1631, 1425, 1377 | 3.36 (m, 4H), 2.17 (s, 3H), 2.09 (t, 2H, J = 7.5), 1.46 (m, 2H), 1.29 (m, 2H), 0.89 (t, 3H, J = 7.2) | 189.5, 173.2, 150.3, 82.7, 40.6 (2C), 28.7, 23.5, 22.5, 13.7, 10.0 | 229 (MH⁺), 228 (M⁺), 200, 158, 143, 130 |
| 2b      | 2962, 1761, 1621, 1372, 1319 | 3.35 (m, 4H), 2.24 (s, 3H), 1.22 (s, 9H) | 188.5, 169.3, 156.8, 82.7, 40.7 (2C), 32.7, 28.0 (3C), 10.9 | 229 (MH⁺), 248 (M⁺), 220, 191, 115 |
| 2c      | 1756, 1624, 1448, 1420, 1368, 1302 | 7.69 (m, 2H), 7.38 (m, 3H), 3.43 (m, 4H), 2.50 (s, 3H) | 187.7, 170.5, 144.6, 129.4, 129.0, 128.8 (2C), 127.5 (2C), 83.9, 40.9 (2C), 11.7 | 249 (MH⁺), 228 (M⁺), 200, 136 |
| 2d      | 2964, 1758, 1621, 1476, 1363 | 6.82 (dd, 1H, J = 17.5, 11.1), 6.33 (d, 1H, J = 17.5), 5.82 (d, 1H, J = 11.1), 3.41 (m, 4H), 1.22 (s, 9H) | 189.9, 160.8, 155.6, 128.3, 125.1, 79.9, 40.6 (2C), 33.5, 28.3 (3C) | 241 (MH⁺), 240 (M⁺) |
| 2e      | 2955, 2929, 1766, 1620, 1459, 1350 | 4.24 (s, 3H), 3.39 (m, 4H), 2.05 (t, 2H, J = 7.6), 1.47 (m, 2H), 1.28 (m, 2H), 0.87 (t, 3H, J = 7.3) | 186.0, 176.6, 125.8, 78.6, 59.3, 40.6 (2C), 29.5, 22.5, 22.3, 13.7 | 245 (MH⁺), 244 (M⁺), 154, 136 |
| 2f      | 2965, 1760, 1614, 1479, 1353 | 4.29 (s, 3H), 3.49 (m, 2H), 3.35 (m, 2H), 1.14 (s, 9H) | 185.0, 173.4, 125.8, 78.4, 58.8, 40.6 (2C), 31.5, 28.0 (3C) | 245 (MH⁺), 244 (M⁺), 154, 136 |
| 2g      | 1768, 1633, 1601, 1456, 1361 | 7.72 (d, 2H, J = 7.7), 7.34 (t, 2H, J = 7.7), 7.27 (t, 1H, J = 7.7), 4.47 (s, 3H), 3.56 (m, 2H), 3.43 (m, 2H) | 183.7, 173.7, 128.5 (2C), 128.3, 128.2, 127.0 (2C), 122.9, 79.5, 59.6, 40.8 (2C) | 264 (M⁺), 236, 221, 165, 121 |
| 2h      | 2956, 2222, 1771, 1614, 1456, 1360 | 4.37 (s, 3H), 3.38 (m, 4H), 2.31 (t, 2H, J = 7.1), 1.50 (m, 2H), 1.38 (m, 2H), 0.89 (t, 3H, J = 7.3) | 183.4, 180.1, 106.1, 95.0, 79.6, 67.1, 61.1, 40.8 (2C), 30.2, 21.9, 19.1, 13.5 | 268 (M⁺), 240, 225, 183, 170 |

* Exact masses with maximum deviation of ± 0.0008 were obtained.

MS (CI): m/z (%) = 187 (M⁺, 94), 186 (M⁺, 77), 158 (100), 127 (65). HRMS (CI): m/z calcd for C₁₈H₂₀S₂: 316.1073. Found: 316.1067.

**Ketenedioacetales 6; General Procedure**

Method A:

Phenyllithium (2.0 M, 0.75 mL, 1.5 mmol) was added dropwise to a solution of the 1,3-dithianol 2 (1.0 mmol) in anhyd THF (5 mL) at −78°C. The resulting solution was stirred at −78°C for 30 min and then quenched with sat. NaHCO₃ (5 mL) and Et₂O (5 mL). The aqueous layer was separated and extracted with Et₂O (2 × 5 mL). The combined organic layers were washed with brine (10 mL) and dried. Removal of the solvent in vacuo followed by flash chromatography (hexanes–EtOAc) provided the product.

**Silylenoethers 7; General Procedure**

Method B:

An organolithium reagent (1.5 mmol) was added dropwise to a solution of the 1,3-dithianol 2 (1.0 mmol) in anhyd THF (5 mL) at −78°C. The resulting solution was stirred at −78°C for 30 min, then chlorotrimethylsilane (0.25 mL, 2.0 mmol) was introduced. The re-

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A solution of 2,3-dimethyl-5,8-dithiaspiro[3.4]oct-2-ene-1-ol (8) in anhyd THF (2 mL) at –5 °C was treated with disobutylaluminum hydride (1 M in hexanes, 1.1 mL, 1.1 mmol) for 10 min. The reaction was monitored by thin-layer chromatography. After cooling to 0 °C, sat. sodium potassium tartrate (10 mL) and Et₂O (10 mL) were added. The resulting mixture was vigorously stirred for 30 min. After cooling the solution to r.t., the solvent was removed in vacuo followed by flash chromatography (hexanes–EtOAc, 8:1) to provide the colorless oil (165 mg, 88% yield).

**Synthesis and Subsequent Ring Opening of Cyclobutene Ethylenedithioacetals**

**Table 4 Spectroscopic Data** for 3-[1,3]Dithiolan-2-ylidine-1-phenyl-but-1-ones 6 and (3-[1,3]Dithiolan-2-ylidine-but-1-enyloxy)-trimethylsilanes 7 Prepared

| Product | IR (cm⁻¹) | ¹H NMR (CDCl₃; 500 MHz) δ (ppm), J (Hz) | ¹C NMR (CDCl₃; 125 MHz) δ (ppm) | MS m/z |
|---------|-----------|----------------------------------------|---------------------------------|------|
| 6a      | 2973, 2928, 1682, 1596, 1447, 1217 | 8.00 (m, 2H), 7.53 (m, 1H), 7.42 (m, 2H), 4.37 (q, 1H, J = 6.7), 3.45 (m, 2H), 3.40 (m, 2H), 1.60 (s, 3H), 1.28 (d, 3H, J = 6.7) | 205.0, 136.6, 132.9, 131.8, 128.5 (2C), 128.3 (2C), 122.4, 50.3, 38.0, 37.9, 18.0, 13.8 | 265 (MH⁺), 264 (M⁺) |
| 6b      | 2956, 2929, 1683, 1596, 1448, 1212 | 8.02 (m, 2H), 7.53 (m, 1H), 7.44 (m, 2H), 4.29 (t, 1H, J = 7.2), 3.55 (s, 3H), 3.37 (m, 4H), 2.06 (m, 1H), 1.86 (m, 1H), 1.56 (m, 4H), 0.91 (t, 3H, J = 7.0) | 198.3, 142.9, 137.0, 132.9, 128.5 (2C), 128.2 (2C), 124.6, 59.4, 53.6, 38.1, 37.8, 29.8, 28.9, 22.8, 14.0 | 323 (MH⁺), 322 (M⁺), 197, 161 |
| 6c      | 2955, 2932, 1687, 1594, 1446, 1364 | 8.03 (m, 2H), 7.53 (m, 1H), 7.45 (m, 2H), 4.28 (s, 1H), 3.49 (s, 3H), 3.36 (m, 4H), 1.13 (s, 9H) | 198.8, 143.4, 138.8, 132.7, 128.5 (2C), 128.0 (2C), 126.8, 61.0, 59.1, 38.1, 37.9, 28.8 (3C) | 323 (MH⁺), 322 (M⁺) |
| 7a      | 2956, 2930, 1625, 1606, 1253, 1102 | 3.46 (s, 3H), 3.33 (m, 2H), 3.20 (m, 2H), 2.20 (m, 1H), 2.07 (m, 1H), 1.64 (m, 1H), 1.39 (m, 1H), 1.29 (m, 2H), 1.17 (s, 9H), 0.89 (t, 3H, J = 7.3), 0.27 (s, 9H) | 154.9, 143.4, 119.8, 116.3, 55.4, 37.7, 37.4, 35.2, 34.5, 29.9, 29.8 (3C), 23.1, 14.0, 1.2 (3C) | 375 (MH⁺), 374 (M⁺), 343, 289, 189 |
| 7b      | 2954, 2929, 1565, 1280, 1253, 1082 | 6.60 (dd, 1H, J = 17.3, 11.1), 5.43 (d, 1H, J = 17.3), 5.11 (d, 1H, J = 11.1), 3.44 (s, 3H), 3.33 (m, 2H), 3.23 (m, 2H), 1.22 (s, 9H), 0.30 (s, 9H) | 151.1, 141.8, 134.8, 125.5, 118.3, 115.7, 55.3, 37.7, 37.6, 35.2, 29.7 (3C), 1.8 (3C) | 345 (MH⁺), 344 (M⁺), 287, 240, 227 |
| 7c      | 2955, 2929, 1624, 1593, 1252, 1108 | 7.41 (m, 2H), 7.23 (m, 3H), 3.40 (s, 3H), 3.19 (m, 3H), 2.95 (m, 1H), 1.30 (s, 9H), 0.02 (s, 9H) | 152.7, 142.7, 139.3, 128.5 (2C), 127.7, 127.1 (2C), 121.8, 116.2, 55.4, 37.5, 37.2, 35.0, 29.5 (3C), 0.9 (3C) | 395 (MH⁺), 394 (M⁺) |
| 7d      | 2956, 2928, 1598, 1252, 1140, 1108 | 7.42 (m, 2H), 7.23 (m, 3H), 3.60 (s, 3H), 3.32 (m, 2H), 2.90 (m, 2H), 2.29 (t, 2H, J = 7.8), 1.45 (m, 2H), 1.36 (m, 2H), 0.92 (t, 3H, J = 7.2), 0.05 (s, 9H) | 151.3, 142.0, 138.8, 128.2 (2C), 127.6, 127.3 (2C), 117.8, 117.5, 56.1, 37.3, 37.1, 29.7, 29.2, 23.1, 14.0, 0.5 (3C) | 394 (M⁺), 323, 217, 191, 136 |
| 7e      | 2955, 2931, 2222, 1587, 1250, 1082 | 3.51 (s, 3H), 3.26 (m, 4H), 2.30 (t, 2H, J = 6.8), 1.47 (m, 4H), 1.18 (s, 9H), 0.90 (t, 3H, J = 7.2), 0.30 (s, 9H) | 142.2, 135.8, 128.7, 120.6, 91.4, 78.5, 55.6, 37.7, 37.6, 35.3, 30.6, 29.1 (3C), 21.9, 18.9, 13.6, 0.7 (3C) | 399 (MH⁺), 398 (M⁺), 383, 341, 202 |

*Exact mass values with maximum deviation ± 0.0008 were obtained.*
chromatography (hexanes–EtOAc, 15:1) to give a colorless oil (43 mg, 23% yield). The product has to be stored in a refrigerator.

IR (film): ν = 2973, 2931, 2807, 2714, 1720, 1597, 1421, 1384, 1279, 1149, 1063 cm⁻¹

1H NMR (500 MHz): δ = 6.26 (s, 1H), 4.84 (s, 1H), 3.70 (m, 2H), 3.61 (m, 1H), 3.48 (m, 1H), 2.00 (s, 3H), 1.81 (s, 3H), 1.00 (s, 9H), 0.37 (s, 3H), 0.26 (s, 3H).

13C NMR (125 MHz): δ = 138.6, 129.5, 123.5, 121.5, 37.6, 37.4, 25.9, 25.7, 23.5, 18.6, 10.5, 8.4, -4.3, -4.8.

MS (CI): m/z (%) = 431 (MH⁺, 7), 373 (77), 302 (97%), 245 (43), 234 (45), 217 (100), 184 (41), 171 (29), 143 (63), 127 (34).

HRMS (CI): m/z calcld. for C₁₈H₂₆O₄S₂Si ((MH⁺): 431.1317. Found: 431.1315.

Preparation of the Cycloadduct 13 with Maleic Anhydride

A solution of silylenol ether 11 (121 mg, 0.4 mmol) and maleic anhydride (43 mg, 0.44 mmol) in dry toluene (2 mL) was refluxed for 24 h. The solvent was removed in vacuo and the crude product was purified by flash chromatography (hexanes–EtOAc, 10:1) to give a colorless oil (83 mg, 52% yield).

IR (film): ν = 2952, 2928, 2857, 1864, 1779, 1472, 1282, 1251, 1231, 1090, 1054, 996, 952, 849, 839, 776, 733 cm⁻¹

1H NMR (500 MHz, CD₂Cl₂): δ = 4.20 (d, 1H, J = 4.8 Hz), 3.30 (m, 2H), 3.05 (d, 1H, J = 11.0 Hz), 2.87 (m, 1H), 2.59 (m, 1H), 2.45 (dd, 1H, J = 11.0, 4.8 Hz), 1.79 (s, 3H), 1.48 (s, 3H), 0.99 (s, 9H), 0.16 (s, 3H), 0.03 (s, 3H).

13C NMR (125 MHz, CD₂Cl₂): δ = 169.4 (2C), 136.5, 130.4, 68.6, 65.8, 53.2, 48.5, 42.5, 42.4, 26.2 (3C), 19.8, 18.4, 15.4, -4.1, -5.1.

MS (CI): m/z (%) = 401 (MH⁺, 77), 357 (24), 343 (67), 299 (100), 269 (35), 239 (60), 234 (48), 225 (37), 208 (28), 165 (53), 135 (23).

HRMS (CI): m/z calcld. for C₁₈H₂₆O₄S₂Si: 400.1198. Found: 400.1196.

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