

Palladium-catalyzed carbonylation of 2-butyne-1,4-diol derivatives: formation of fulgide or lactone

Jitsuo Kiji^{a,*}, Yuuichi Kondou^a, Masahiro Asahara^a,
Yasushi Yokoyama^b, Toshiya Sagisaka^b

^a Department of Materials Science, Faculty of Engineering, Tottori University, Koyama Minami, 680-8552 Tottori, Japan

^b Department of Advanced Materials Chemistry, Graduate School of Engineering, Yokohama National University, 240-8501 Yokohama, Japan

Received 10 October 2002; accepted 18 November 2002

Abstract

The carbonylation of the substituted 2-butyne-1,4-diols, RArC(OH)≡C(OH)ArR (Ar, 2,5-dimethylthienyl; R, alkyl) was studied by using Pd(OAc)₂/I₂ as the catalyst in benzene under pressure. The diols bearing methyl or isopropyl group as the alkyl substituent gave photochromic fulgide (**4a** or **4b**). On the other hand, the carbonylation of the diol bearing bulky *t*-butyl group did not afford the corresponding fulgide but lactone **5**, together with butatriene **6**.

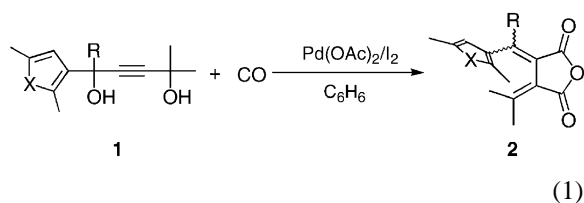
© 2002 Elsevier Science B.V. All rights reserved.

Keywords: Palladium catalyst; Carbonylation; Propargylic alcohol; Photochromic fulgide; Lactonization

1. Introduction

Propargylic compounds (substituted 2-alkynyl compounds) undergo several types of transformations promoted by palladium complexes [1–3]. Among them, carbonylation is considered as an important reaction in organic synthesis and has been studied extensively [4–11]. Photochromic fulgides such as **2** have attracted attention as a functional molecule, which can be applied to a photochemical memory device [12–14]. In general, fulgides have been synthesized by the conventional method based on the Stobbe condensation [15]. Previously, the authors have proposed a convenient synthetic route to sterically congested fulgides (**2**), which are hardly synthesized by the Stobbe condensation, by the palladium-catalyzed carbonylation

of heterocycle-substituted 2-butyne-1,4-diol derivatives **1** (Eq. (1)) [16,17].

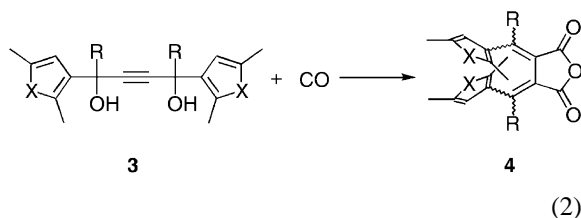


(a, X = S, R = CH₃; b, X = O, R = CH₃; c, X = S, R = *i*-C₃H₇; d, X = S, R = *t*-C₄H₉).

Recently, we applied this method to the synthesis of bisfuryl or bithienylfulgides **4a** (Eq. (2)). Bisthienylfulgide **4a** thus obtained shows the diastereoselective photocyclization [18]. The preliminary results have already been reported [19]. In the present study, we carried out the carbonylation of more sterically congested, thienyl-substituted diols **3c** and **3d** bearing isopropyl and *t*-butyl as R, respectively, since the fulgide

* Corresponding author. Present address: 1-59 Mihagino, Tottori 689-0202, Japan. Tel.: +81-857-590250; fax: +81-857-590250.

of this type, which is modified by the bulky substituent, was expected to show a high quantum yield of the photocyclization [20]. It has now been found that there is a great difference between the isopropyl and *t*-butyl derivatives in the carbonylation reaction. The present paper is concerned with more details on the carbonylation of bisthienyl-substituted diols **3**.



(a, X = S, R = CH₃; b, X = O, R = CH₃; c, X = S, R = *i*-C₃H₇; d, X = S, R = *t*-C₄H₉).

2. Experimental

2.1. General

Solvents were purified by conventional methods, stored, and used under argon atmosphere. Other commercially available reagents were used without further purification. Spectral data were recorded by the following instruments: IR, Perkin-Elmer 1600 FT-IR; NMR, JEOL FNM-ECP 500 MHz MNR; GC/MS, HITACHI M-4100 MS/MS; UV, JASCO V-550 UV-Vis spectrometers. X-ray structural analysis was performed by a RIGAKU RAXIS-IV refractometer.

2.2. Synthesis of **3c**

All the procedures were performed under an argon atmosphere. In a three-necked flask, were placed 28 ml of 1.53 molar hexane solution of BuLi (42.8 mmol) and 5 ml of THF. The flask was cooled at -78°C and then 1.81 g (13.8 mmol) of trichloroethylene in 4 ml of THF was added dropwise. The temperature was gradually raised to room temperature and the mixture was stirred for 2 h. The flask was cooled again at -78°C and 2.56 g (14 mmol) of 3-isobutyl-2,5-dimethylthiophene in 3 ml of THF was added dropwise. The temperature was gradually raised to room temperature and the mixture was stirred

overnight. After aqueous NH₄Cl was added, the aqueous layer was extracted with ether. The ethereal solution was dried over Na₂SO₄. After evaporating ether, the oily viscous residue was chromatographed on 30 ml of Wakogel C-200 (75–150 μm), using hexane (20 ml), hexane/toluene (1/1, 160 ml), toluene (160 ml), chloroform (370 ml), and methanol (70 ml) as the eluent in this order. The eluate with hexane/toluene gave 0.53 g (10% yield) of **3c**. ¹H NMR (CDCl₃) δ (ppm) 0.95 (6H, d, $J = 6.89$ Hz), 1.07 (6H, d, $J = 6.89$ Hz), 2.19 (2H, m) 2.36 (6H, s), 2.48 (6H, s), 6.71 (2H, s).

2.3. Synthesis of **3d**

The reaction was carried out in a similar manner as described above using 27.2 mmol of BuLi, 9.13 mmol of trichloroethylene, and 7.85 mmol of 3-pyvaroyl-2,5-dimethylthiophene. A solid separated from the ethereal solution. Recrystallization from hexane gave **3d** (1.46 g) in a 39% yield. ¹H NMR (CDCl₃) δ (ppm) 1.08 (18H, s), 2.34 (6H, s), 2.51 (6H, s), 6.67 (2H, s).

2.4. Carbonylation of **3c** (synthesis of fulgide **4c**)

In a 40 ml stainless steel autoclave 1.20 g (2.4 mmol) of diol **3c**, 27 mg of Pd(OAc)₂, 30 mg of I₂ and 10 ml of benzene were placed. Carbon monoxide was charged up to 95 atm and the autoclave was heated at 90°C for 15 h with magnetic stirring. The solution was filtered through Wakogel C-200. After evaporating benzene under reduced pressure, the residue was chromatographed on Wakogel C-200 (30 ml) with 100 ml of hexane, 180 ml of hexane/toluene (2/1), 180 ml of hexane/toluene (1/1), 120 ml of hexane/toluene (1/2), 120 ml of toluene, and 80 ml of methanol in this order. The eluate with hexane/toluene (2/1) gave the photochromic **4c** (0.24 g, 23% yield) of an isomeric mixture, which was subjected further to flash chromatography (Merck Kieselgel 60) to separate the isomers. The EE isomer (both pairs of thienyl and carbonyl groups are in the E configuration) was isolated in a 1.5% yield based on **3c**: mp, 161–163 $^{\circ}\text{C}$; HRMS, calc. for C₂₄H₂₈O₃S₂ 428.1480, obs. 428.1479; ¹H NMR (CDCl₃) δ (ppm) 0.58 (6H, d, $J = 6.93$ Hz), 1.20 (6H, d, $J = 6.93$ Hz), 2.07 (6H, s), 2.20 (6H, s), 4.05 (2H, m), 5.70 (2H, s); IR

(KBr), λ_{co} (cm^{-1}) 1756, 1810. The other isomers have not yet been isolated in a pure form.

2.5. Carbonylation of **3d** (synthesis of lactone **5**)

The typical procedure is as follows. The carbonylation was performed under 70 atm at 90 °C for 20 h using 0.42 g (1.00 mmol) of **3d** and 5 mol% of the catalyst ($\text{Pd}(\text{OAc})_2/\text{I}_2$) in 10 ml of benzene. In a similar manner, the products were isolated by column chromatography. A solid isolated from the eluate with hexane (60 ml) was recrystallized from methanol to give light brown needles **6** (70 mg): mp, 145–149 °C; HRMS, calc. for $\text{C}_{24}\text{H}_{32}\text{S}_2$ 384.1947, obs. 384.1938; ^1H NMR (CDCl_3), δ (ppm) 1.09 (18H, s), 2.33 (6H, s), 6.58 (2H, s), 2.40 (6H, s).

The solid mass separated from the eluate with hexane/toluene (2/1, 150 ml) was chromatographed again, after removing methanol-insoluble **6**, which was included as colorless needles in a small amount. The eluate with hexane/toluene (2/1) gave crystals, which were recrystallized from methanol to give **5** as pale yellow plates (70 mg): mp, 176.0–178.5 °C; HRMS, calc. for $\text{C}_{25}\text{H}_{32}\text{O}_2\text{S}_2$ 428.1845, obs. 428.1845; ^1H NMR (CDCl_3), δ (ppm) 1.06 (9H, s), 1.33 (9H, s), 1.53 (3H, s), 2.15 (3H, s), 2.40 (3H, s), 2.43 (3H, s), 6.23 (1H, s); IR (KBr), λ_{co} (cm^{-1}) 1747.

2.6. X-ray crystallographic analysis of **5**

The single crystals recrystallized from methanol were subjected to crystallographic analysis. The intensity data were collected at 173 K on a Rigaku RAXIS-IV imaging plate area detector with graphite-monochromated Mo $\text{K}\alpha$ ($\lambda = 0.71070 \text{ \AA}$) radiation from a rotating-anode generator operating at 50 kV and 100 mA. A total of 24 oscillating images, each oscillating 6° and exposed for 20 min, were collected for **5**. The data were collected for Lorentz and polarization effect. A correction for secondary extinction was applied (coefficient = $4.31941 e^{-0.5}$). The structure was solved by direct methods (SIR92) [21] and expanded using Fourier techniques (DIRDIF94) [22]. The non-hydrogen atoms were refined anisotropically. Hydrogen atoms were included at calculated positions, but not refined. All calculations were performed using *teXsan* crystallographic software package of Molecular Structure Corporation. Their crystal data

Table 1
Crystallographic data for **5**

| Crystallographic data | 5 |
|---|--|
| Chemical formula | $\text{C}_{25}\text{H}_{32}\text{O}_2\text{S}_2$ |
| Formula weight | 428.65 |
| Crystal dimensions (mm^3) | $0.5 \times 0.5 \times 0.3$ |
| Crystal system | Triclinic |
| Space group | $P1$ (no. 2) |
| μ (Mo $\text{K}\alpha$) (cm^{-1}) | 2.51 |
| R | 0.073 |
| R_w | 0.099 |
| GOF | 1.93 |
| Unit-cell dimensions | |
| a (\AA) | 11.085(1) |
| b (\AA) | 12.4345(9) |
| c (\AA) | 9.2370(5) |
| a ($^\circ$) | 103.481(5) |
| b ($^\circ$) | 110.183(6) |
| g ($^\circ$) | 94.938(4) |
| V (\AA^3) | 1142.6200 |
| D_{calcd} (g cm^{-3}) | 1.246 |
| Temperature (K) | 263 |
| Z | 2 |
| No. of variables | 263 |
| No. of reflections | 4542 |
| No. of reflections used ($I > 3.00\sigma(I)$) | 4058 |
| $\rho_{\text{max}}/\rho_{\text{min}}$ ($e^- \text{\AA}^{-3}$) | 0.52/−0.65 |

and experimental details were listed in Table 1. Further details of the crystallographic data have been deposited to the Cambridge Crystallographic Data Centre.¹

3. Results and discussion

Diols **3c** and **3d** were prepared by the reaction of the corresponding alkyl thienyl ketone with dilithium diacetylide, which was prepared in situ by the reaction of butyllithium with trichloroethylene at −78 °C [23].

The carbonylation was performed in benzene under pressure at 90 °C, using equimolar amounts of $\text{Pd}(\text{OAc})_2$ and I_2 as the catalyst. It is to be noted that the presence of I_2 is essential for the carbonylation

¹ Crystallographic data for the structural analysis has been deposited with the Cambridge Crystallographic Data Centre, CCDC No. 196144 for **5**. Copies of this information may be obtained free of charge from: The Director CCDC, 12 Union Road, Cambridge CB2 1EZ, UK. Fax. +44-1223-336-033. E-mail address: deposit@ccdc.cam.ac.uk or web site: <http://www.ccdc.cam.ac.uk>.

of propargyl alcohols of this type [5]. After evaporating benzene, the residue showed medium and strong IR absorptions at 1809 and 1763 cm^{-1} , respectively. The products were subjected to TLC analysis. The formation of photochromic fulgides was confirmed by irradiating the TLC plate with UV light. When the fulgides were formed, a violet spot was observed on the plate and slow fading was noted on standing. Thus, diol **3c** was dicarbonylated and photochromic fulgide **4c** was obtained as a mixture of stereoisomers in a ca. 23% yield by column chromatography. The mixture was further subjected to flash chromatography. The ZZ isomer, in which both the pairs of the thienyl and the carbonyl groups are in the Z configuration, was isolated from the mixture. The separation of the other stereoisomers in a pure form has been unsuccessful. These results imply that diol **3c** is subjected to the normal carbonylation, which is widely observed in the carbonylation of propargylic alcohols and 2-butyne-1,4-diols [3]; namely, the addition of carbon monoxide occurs at the acetylenic carbon atom, followed by the bond migration and elimination of the hydroxyl group. This isomer (λ_{max} , 396 nm) cyclized to afford the colored form (λ_{max} , 511 nm) and showed photochromism.

As noted above, the yield of the fulgide as the carbonylation product was moderate. In the procedure of the column chromatography, the last two eluates with CHCl_3 and CH_3OH possessed a strong IR absorption around 1680 cm^{-1} due to an acid [16]. Therefore, the formation of the diacid, i.e. the hydrolyzed product of the fulgide, is a cause of the moderate yield.

Similarly, the carbonylation of **3d** was carried out at 90 °C. The formation of the photochromic fulgide was not confirmed by TLC. The carbonylation product isolated by column chromatography showed a strong carbonyl absorption at 1747 cm^{-1} , which is due to a monocarbonylated product. Its ^1H NMR spectrum showed eight singlet signals, which are divided into three groups; (i) *t*-butyl, 1.06 and 1.33 ppm, (ii) methyl, 1.53, 2.15, 2.40, and 2.43 ppm, and (iii) thienyl, 6.23 and 6.74 ppm. These spectroscopic data indicated that the carbonylation of **3d** followed quite a different course from that of **3c**. That is to say, the dicarbonylation of **3d** did not take place. The structure of **5** was established by the X-ray structural analysis (Fig. 1 and Table 2).

Table 2
Bond distances and angles of **5**

| Bond distances (Å) | | Bond angles (°) | |
|--------------------|----------|-------------------|-----------|
| S(1)–C(6) | 1.774(2) | C(6)–S(1)–C(7) | 90.27(10) |
| S(1)–C(7) | 1.850(2) | C(16)–S(2)–C(19) | 93.4(1) |
| S(2)–C(16) | 1.732(2) | C(1)–O(2)–C(9) | 112.1(2) |
| S(2)–C(19) | 1.721(3) | O(1)–C(1)–O(2) | 121.0(2) |
| O(1)–C(1) | 1.209(3) | O(1)–C(1)–C(2) | 131.9(2) |
| O(2)–C(1) | 1.361(3) | O(2)–C(1)–C(2) | 107.1(2) |
| O(2)–C(9) | 1.470(2) | C(1)–C(2)–C(3) | 139.0(2) |
| C(1)–C(2) | 1.483(3) | C(1)–C(2)–C(8) | 108.3(2) |
| C(2)–C(3) | 1.484(3) | C(3)–C(2)–C(8) | 112.7(2) |
| C(2)–C(8) | 1.343(3) | C(2)–C(3)–C(4) | 105.1(2) |
| C(3)–C(4) | 1.366(3) | C(2)–C(3)–C(10) | 124.3(2) |
| C(3)–C(10) | 1.507(3) | C(4)–C(3)–C(10) | 130.5(2) |
| C(4)–C(5) | 1.454(3) | C(3)–C(4)–C(5) | 137.5(2) |
| C(4)–C(7) | 1.520(3) | C(3)–C(4)–C(7) | 111.0(2) |
| C(5)–C(6) | 1.343(3) | C(5)–C(4)–C(7) | 110.5(2) |
| C(6)–C(14) | 1.492(3) | C(4)–C(5)–C(6) | 113.0(2) |
| C(7)–C(8) | 1.505(3) | S(1)–C(6)–C(5) | 113.7(2) |
| C(7)–C(15) | 1.552(3) | S(1)–C(6)–C(14) | 118.2(2) |
| C(8)–C(9) | 1.508(3) | C(5)–C(6)–C(14) | 128.1(2) |
| C(9)–C(17) | 1.537(3) | S(1)–C(7)–C(4) | 102.6(1) |
| C(9)–C(22) | 1.578(3) | S(1)–C(7)–C(8) | 122.8(1) |
| C(10)–C(11) | 1.538(3) | S(1)–C(7)–C(15) | 108.4(1) |
| C(10)–C(12) | 1.541(3) | C(4)–C(7)–C(8) | 102.4(2) |
| C(10)–C(13) | 1.536(3) | C(4)–C(7)–C(15) | 110.4(2) |
| C(16)–C(17) | 1.382(3) | C(8)–C(7)–C(15) | 109.4(2) |
| C(16)–C(20) | 1.488(4) | C(2)–C(8)–C(7) | 107.7(2) |
| C(17)–C(18) | 1.428(3) | C(2)–C(8)–C(9) | 111.0(2) |
| C(18)–C(19) | 1.362(4) | C(7)–C(8)–C(9) | 139.8(2) |
| C(19)–C(21) | 1.491(4) | O(2)–C(9)–C(8) | 101.5(2) |
| C(22)–C(23) | 1.536(3) | O(2)–C(9)–C(17) | 108.1(2) |
| C(22)–C(24) | 1.538(3) | O(2)–C(9)–C(22) | 105.4(2) |
| C(22)–C(25) | 1.538(3) | C(8)–C(9)–C(17) | 111.0(2) |
| | | C(8)–C(9)–C(22) | 113.8(2) |
| | | C(17)–C(9)–C(22) | 115.7(2) |
| | | C(3)–C(10)–C(11) | 109.0(2) |
| | | C(3)–C(10)–C(12) | 108.7(2) |
| | | C(3)–C(10)–C(13) | 112.3(2) |
| | | C(11)–C(10)–C(12) | 110.1(2) |
| | | C(11)–C(10)–C(13) | 108.4(2) |
| | | C(12)–C(10)–C(13) | 108.4(2) |
| | | S(2)–C(16)–C(17) | 110.2(2) |
| | | S(2)–C(16)–C(20) | 117.0(2) |
| | | C(17)–C(16)–C(20) | 132.7(2) |
| | | C(9)–C(17)–C(16) | 122.6(2) |
| | | C(9)–C(17)–C(18) | 125.4(2) |
| | | C(16)–C(17)–C(18) | 111.8(2) |
| | | C(17)–C(18)–C(19) | 114.8(2) |
| | | S(2)–C(19)–C(18) | 109.7(2) |
| | | S(2)–C(19)–C(21) | 122.2(2) |
| | | C(18)–C(19)–C(21) | 128.1(3) |

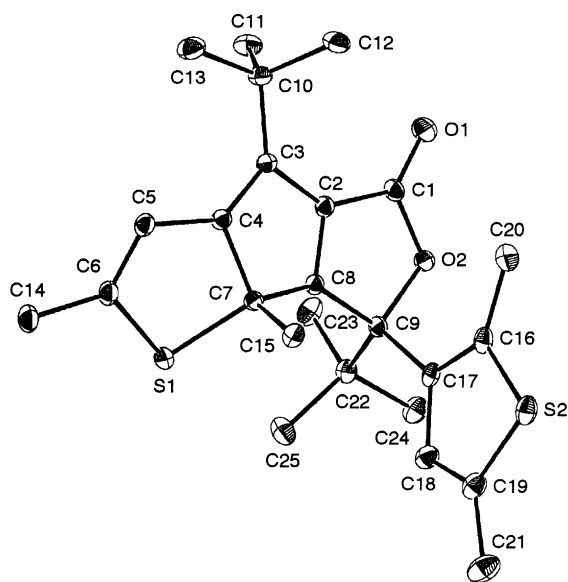
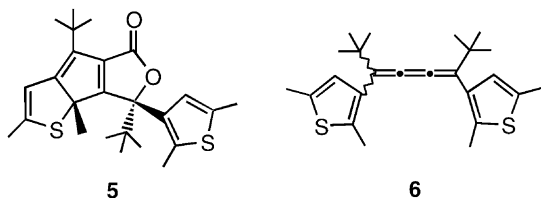


Fig. 1. Crystal structure of **5** drawn at the 30% probability level. All hydrogen atoms are omitted for clarity.

It has been revealed that five-membered ring lactone **5** is the carbonylation product of this reaction. It was also found that a butatriene derivative (**6**) was formed in a comparable yield.

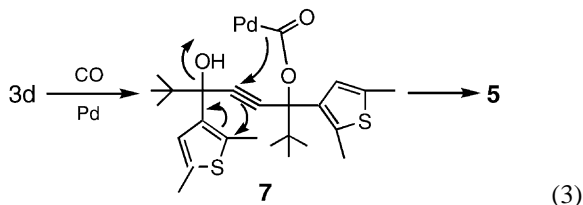


This finding implies the following fact, that is, in the carbonylation of more bulky **3d** around the acetylenic carbon atoms, the steric congestion is so large that the addition of carbon monoxide does not occur to the triple bond.

To study the carbonylation of **3d** in further detail, the reaction was performed under different conditions. At 70 and 90 °C, lactone **5** was formed in 8 and 16% yields, respectively and **6** in 16 and 18% yields, respectively. Even at 90 °C, **3d** was recovered in a small amount after 12 h. At 120 °C, however, the carbonylation did not take place, instead, the decomposition of **3d** was observed. At lower temperature, a consid-

erable amount of the diol was recovered unchanged. Therefore, the range of 70–90 °C looks like a favorable choice for the formation of **5**. Thus, the photochromic compound was not obtained at all from **3d**.

The formation of lactone **5** can be explained as follows. The steric congestion around the acetylenic carbon atoms is so large that the addition of carbon monoxide does not occur to the acetylenic carbon atoms. This is a marked contrast between **3c** and **3d**, i.e. the hydroxyl group of **3d** is more susceptible to the carbonylation than the triple bond. In this case, the first step is the formation of an alkoxy palladium species **7**, which then brings about the cascade ring closing reaction after the insertion of carbon monoxide into the Pd–O bond as shown in Eq. (3).



Similar conceivable intermediate to **7** for the lactonization of unsaturated alcohols has been proposed [24].

4. Conclusion

The limitation of the method, in which, the palladium-catalyzed carbonylation of the substituted 2-butyne-1,4-diols is applied to the synthesis of photochromic dithienylfulgides **4**, is that the dicarbonylation of the diol **3** bearing bulky substituent such as *t*-butyl as R does not occur. Instead, the monocarbonylation, followed by cascade ring closing reaction, takes place to afford lactone.

Acknowledgements

The authors are grateful to Dr. Kayoko Saiki of Kobe Pharmaceutical University for the measurement of mass spectra and also indebted to Prof. Kohei Tamao of Institute for Chemical Research, Kyoto University, for the use of Rigaku RAXIS-IV diffractometer.

References

- [1] J. Tsuji, *Reagents and Catalysts: Innovations in Organic Synthesis*, Wiley, Chichester, 1995, 453 pp.
- [2] J. Tsuji, J. Kiji, in: M. Beller, Bolm (Eds.), *Transition Metals for Organic Synthesis*, vol. 1, Wiley-VCH, Weinheim, 1998, p. 68.
- [3] J. Tsuji, T. Mandai, *Angew. Chem. Int. Ed. Engl.* 34 (1995) 3001.
- [4] J. Tsuji, T. Nogi, *Tetrahedron* 25 (1969) 4099.
- [5] J. Kiji, H. Konishi, T. Okano, S. Kometani, A. Iwasa, *Chem. Lett.* (1987) 313.
- [6] J. Tsuji, T. Sugiura, I. Minami, *Tetrahedron Lett.* 27 (1986) 731.
- [7] Y. Inoue, K. Ohuchi, I.-F. Yen, S. Imaizumi, *Bull. Chem. Soc. Jpn.* 62 (1989) 3518.
- [8] B. Gabriele, M. Costa, G. Salerno, G.P. Chiusoli, *J. Chem. Soc., Chem. Commun.* (1992) 1007.
- [9] B. Gabriele, M. Costa, G. Salerno, G.P. Chiusoli, *J. Chem. Soc., Chem. Commun.* (1994) 1429.
- [10] T.H. Hu, A. Orita, H. Alper, *J. Org. Chem.* 58 (1993) 6956.
- [11] J. Kiji, T. Okano, E. Fujii, J. Tsuji, *Synlett.* (1997) 869.
- [12] H.G. Heller, *Chem. Ind. (London)* (1978) 193.
- [13] Y. Yokoyama, S. Uchida, Y. Yokoyama, Y. Sugawara, Y. Kurita, *J. Am. Chem. Soc.* 118 (1996) 3100.
- [14] Y. Yokoyama, *Chem. Rev.* 100 (2000) 1717.
- [15] H. Stobbe, *Chem. Ber.* 38 (1905) 3673.
- [16] J. Kiji, T. Okano, H. Kitamura, Y. Yokoyama, S. Kubota, Y. Kurita, *Bull. Chem. Soc. Jpn.* 68 (1995) 616.
- [17] S. Uchida, Y. Yokoyama, J. Kiji, T. Okano, H. Kitamura, *Bull. Chem. Soc. Jpn.* 68 (1995) 2961.
- [18] Y. Yokoyama, T. Sagisaka, Y. Yamaguchi, Y. Yokoyama, K. Kiji, T. Okano, A. Takemoto, S.-Y. Mio, *Chem. Lett.* (2000) 220.
- [19] J. Kiji, T. Okano, A. Takemoto, S.-Y. Mio, T. Konishi, Y. Kondou, T. Sagisaka, Y. Yokoyama, *Mol. Cryst. Liq. Cryst.* 344 (2000) 235.
- [20] Y. Yokoyama, T. Goto, T. Inoue, M. Yokoyama, Y. Kurita, *Chem. Lett.* (1988) 1049.
- [21] A. Altomare, M.C. Burla, M. Camalli, M. Cascarano, C. Giacovazzo, A. Guagliardi, G. Polidori, SIR92, *J. Appl. Crystallogr.* 27 (1994) 435.
- [22] P.T. Beurskens, G. Admiraal, G. Beurskens, W.P. Bosman, R. de Gelder, R. Israel, J.M.M. Smits, 1994. DIRDIF94: The DIRDIF-94 Program System, Technical Report of Crystallography Laboratory, University of Nijmegen, The Netherlands.
- [23] (a) S.I. Maghsoodi, Y. Pang, T.J. Barton, *J. Polym. Sci., Part A. Polym. Chem.* 28 (1990) 955;
(b) I. Lin, S.I. Maghsoodi, T.J. Barton, R.K. Meyer, R.E. Bruer, Z.V. Vardeny, X. Wei, A.V. Smith, J. Shinar, *Polym. Prepr.* 35 (2) (1991) 5357.
- [24] B.E. Ali, H. Alper, *J. Org. Chem.* 56 (1991) 5357.