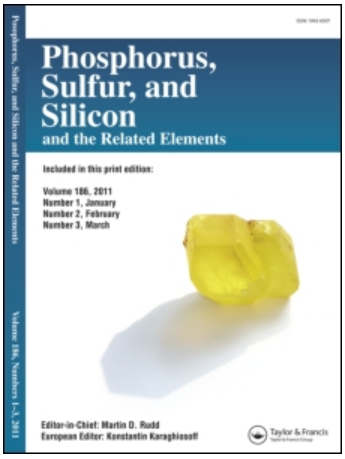


Phosphorus, Sulfur, and Silicon and the Related Elements

Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713618290

One-Pot Synthesis of Sulfides by Reaction of Disulfides with Alkyl Halides in the Presence of Sodium Dithionite

Ri-Yuan Tang[a]; Ping Zhong[a]; Qiu-lian Lin[a]
[a] The School of Chemistry and Material Science, Wenzhou University, Wenzhou, P.R. China

To cite this Article Tang, Ri-Yuan , Zhong, Ping and Lin, Qiu-lian(2007) 'One-Pot Synthesis of Sulfides by Reaction of Disulfides with Alkyl Halides in the Presence of Sodium Dithionite', Phosphorus, Sulfur, and Silicon and the Related Elements, 182: 1, 167 — 174
To link to this Article: DOI: 10.1080/10426500600892479
URL: http://dx.doi.org/10.1080/10426500600892479

Phosphorus, Sulfur, and Silicon, 182:167–174, 2007

ISSN: 1042-6507 print / 1563-5325 online
DOI: 10.1080/10426500600892479

Taylor & Francis
Taylor & Francis Group

One-Pot Synthesis of Sulfides by Reaction of Disulfides with Alkyl Halides in the Presence of Sodium Dithionite

Ri-Yuan Tang
Ping Zhong
Qiu-lian Lin
The School of Chemistry and Material Science, Wenzhou University, Wenzhou, P.R. China

Sodium dithionite-promoted synthesis of unsymmetrical diorganyl sulfides by a reaction of diaryl disulfides with alkyl halides at r.t. has been developed. The advantages offered by this method are operational simplicity, a faster reaction, neutral and mild reaction conditions, and moderate to good yields of products.

Keywords Disulfide; electron transfer; one-pot; sodium dithionite; sulfide

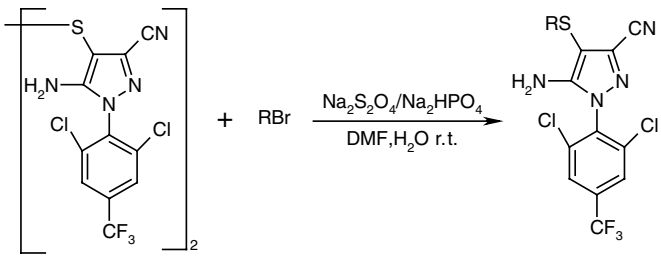
INTRODUCTION

Organic sulfides are useful chemical intermediates in organic synthesis,[1] and a C–S bond can be found in many molecules that are of biological, pharmaceutical, and materials interest.[2] The formation of a C–S bond is usually achieved by the reaction of thiolate or thiol with organic halides. However, these reactions require harsh reaction conditions, and the yields and reaction conditions depend on the solvent, strong basic catalyst, and acidity of thiol.[3] Recently, Yin and Pidgeon[3c] reported a high-yield method for the preparation of unsymmetrical sulfides using very strong basic n-butyllithium. For developing mild reaction conditions, Shah et al.[4] reported a synthesis method of thioethers using CsF–Celite in acetonitrile. Transition metal-mediated alkylation has also been developed for the preparation of thioethers,[5] which is a mild and efficient method. For example, a one-pot and

Received February 26, 2006; accepted June 6, 2006.

This project was supported by the National Natural Science Foundation of China (No. 20572079) and the National Natural Science Foundation of Zhejiang Province (Nos. Y205540 and Y404039). Supported by the Postgraduate Innovation Foundation of Wenzhou University (No. YCX0514).

Address correspondence to Ping Zhong, The School of Chemistry and Material Science, Wenzhou University, Wenzhou, P.R. China. E-mail: zhomgp0512@163.com



1 **2** **3**

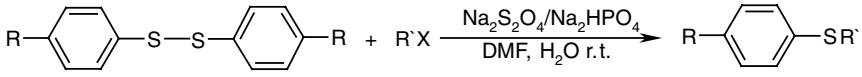
SCHEME 1

base-free conversion of disulfides to sulfides using an in-situ generated organocobalt(III) reagent had been studied.[6] A general and efficient Cu-catalyzed carbon-sulfur bond formation under mild conditions has also been reported.[7] Indium(I) iodide-mediated cleavage of disulfides and subsequent reaction with alkyl halides at r.t., and give high yields.[8]

Although these metal-mediated synthestic methods have made big progress in the synthesis of thioether, the development of a one-step synthetic method using commercial and cheaper reagents under neutral conditions has attracted much attention. Recently we reported the use of sodium dithionite[9] for the synthesis of pyrazolyl alkyl sulfides 3 (Scheme 1).[10] In this article, we demonstrate a further extension of this work together with the application of sodium dithionite for the synthesis of diorganyl sulfides 6 by a reaction of diaryl disulfides 4 with alkyl halides (Scheme 2).

RESULTS AND DISCUSSION

The experimental procedure was very simple. A mixture of alkyl halide and diaryl disulfide in dimethylformamide (DMF) as a 4:l cosolvent with H_2O was stirred in the presence of sodium dithionite at r.t. for a certain period of time (TLC). Usual workup and extraction with ether provided the product.



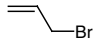
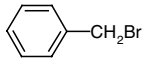
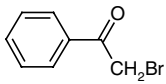
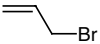
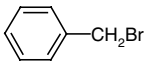
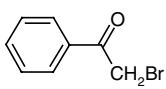
4 **5** **6**

4a: R=NO_2

4b: R=Cl

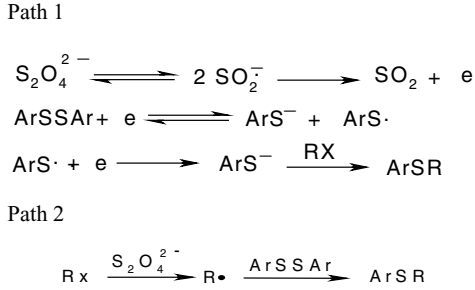
SCHEME 2

TABLE I The Synthesis of Compounds 6a–l

Entry	R	R′X	m.p.(°C)	Reaction Time (h)	Isolated Yield (%)
6a	NO_2	CH_3I	71∼72	0.2	82
6b	NO_2	C_2H_5Br	37∼38	0.3	56
6c	NO_2	*n*-C_3H_7Br	Liquid	0.3	49
6d	NO_2		36∼37	0.25	68
6e	NO_2		118∼119	0.25	73
6f	NO_2		115∼116	0.3	71
6g	Cl	CH_3I	18∼19	3	65
6h	Cl	C_2H_5Br	Liquid	5	48
6i	Cl	*n*-C_3H_7Br	Liquid	5	41
6j	Cl		Liquid	4	53
6k	Cl		49∼50	4	57
6l	Cl		78∼79	5	51

Diverse alkyl halides underwent reactions with diaryl disulfides by this procedure to produce the corresponding alkyl aryl sulfides, respectively, in moderate to good yields. The results are presented in Table I. As evident from Table I, alkyl, allylic, benzyl, benzyl bromides and bromoaceto phenone and methyl iodides could participate in this reaction to form the corresponding products. In general, the reaction of 4b took longer (3–5 h) than 4a (<30 min) by this procedure. However, aryl halides and vinyl halides remained inactive in this reaction; on the other hand, nonaromatic disulfides failed to undergo any reaction by this reaction system. Proton and carbon-13 NMR data is presented here. From the data, we found that the variation of the chemical shifts on the CH_2 connecting the sulfur atom were inagreement with the theoretic variation. When the CH_2 bearing an alkyl, ^1H, and ^{13}C shift move toward the lower magnetic field, while having the p-Π conjugation effect, both shifts move toward the higher magnetic field. For example, the shift change in compounds 6a–f is increasing.

We have speculated that sodium dithionite could serve as an electron source for the cleavage of an S—S bond or the dehalogenation of alkyl halides. Therefore, two reaction pathways for the formation of alkyl



SCHEME 3 Possible reaction pathways for the formation of sulfides.

phenyl sulfides are shown in Scheme 3. This reaction procedure might involve Electronic Transfer (ET). Electron uptake by S—S caused the breaking of the S—S bond to yield a radical (ArS·) and an anion (ArS-). Therefore, the reactions were going through the intermediacy of a radical and an anion, which formed readily by the reaction of an electron released by sodium dithionite with diaryl disulfide. Then the anion (ArS-) was alkylated with alkyl halide (Path 1). Another pathway including the reaction of alkyl radicals, which was generated by the reduction of alkyl halides with sodium dithionite, with aryl disulfides was suggested (Path 2). It was said that sodium dithionite was capable of producing the alkyl free radicals from alkyl halides.[9m–q] Moreover, the SO_2 radical anion, which can release an electron, was readily detected by electron spin resonance in solutions of decomposing S_2O_4,[2–11] so both pathways would be possible.

Theoretical analysis about the reactions of disulfides involving ET processes have been reported.[12–15] S. Antonello et al.[12] suggested that with diaryl disulfides bearing electron-donating or mildly electron-withdrawing groups, the inner reorganization is particularly large, which reflects the significant stretching of the S—S bond experienced by the molecule upon ET. With electron-withdrawing groups, the extent of delocalization of the SOMO (singly occupied MO) onto the aryl system increases, leading to a decrease of the reorganization energy for radical anion formation. From the previous description, we should consider that the stabilization of the formed radicals plays an important role in the reaction. The ArS· radical-bearing electron-withdrawing groups have higher stabilization than that of one bearing electron-donating or mildly electron-withdrawing groups. It is well known that nitro is strong electron-withdrawing group, while the chloro- is a mild one. Thus, the formation of radical from 4a is much more easy than that from 4b. That is why the reaction of 4a took much shorter time than 4b by this procedure.

It can be concluded that a one-pot procedure for the synthesis of alkyl aryl sulfides has been developed. Although the result in this article is not better than that carried out by transition metal,[6,7c,8,16] this work demonstrates the synthetic potential of sodium dithionite and develops a free-base and free-metal synthestic method of aryl alkyl sulfides. The significant advantages offered by this method are operational simplicity, a faster reaction, neutral and mild (r.t.) reaction conditions, and moderate to good yields of products.

EXPERIMENTAL

All melting points were determined with a Thomas-Hoover melting point apparatus. The thermometer was not standardized. ^{1}H spectra were recorded at 300 MHz (^{1}H) and ^{13}C at 75 Mz on a (^{13}C) spectrometer using $CDCl_3$ as a solvent with TMS as the internal standard. J values are given in Hz. IR spectra were measured on a FTIR spectrophotometer as KBr disks. Silica gel 60 GF254 was used for analytical and preparative TLC.

General Procedure for Synthesis of 6a–l

Aryl disulphide **4** (0.3 mmol) was dissolved in dimethylformamide (8 mL); sodium hydrogen phosphate (0.15 g, 0.42 mmol) was dissolved in water (2 mL). The dimethylformamide solution was then introduced into a 50-mL glass reactor; alkyl halide **5** (0.72 mmol) was injected into the mixture, followed by the aqueous solution. Sodium dithionite (0.075 g, 0.42 mmol) was introduced in its turn with stirring. After a certain period of time, water (50 mL) was then added, and this latter mixture was subjected to a careful extraction with a portion of ethyl ether (20×3 mL). The ether phase was separated off. The ether phase was then eluted by water (20×2 mL). Finally, dehydrated $MgSO_4$ and the product were subjected to chromatography on a column of silica gel, eluting with petroleum ether and ethyl acetate. The solution was removed under reduced pressure, giving sulfide **6**.

1-Methylthio-4-nitrobenzene 6a ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.56 (3H, s, SCH_3), 7.28 (2H, dd, JD 1.9, JD 9.0, ArH), 8.14 (2H, dd, JD 1.9, JD 9.0, ArH). ^{13}C NMR (75 MHz, $CDCl_3$) δ 14.8 (1C, SCH_3), 123.9 (2C, Ar), 125.0 (2C, Ar), 144.7 (1C, Ar), 148.8 (1C, Ar). Anal. calcd. for $C_7H_7NO_2S$: C, 49.69; H, 4.17. Found: C, 49.51; H, 4.07.

1-Ethylthio-4-nitrobenzene 6b ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.40 (3H, t, JD 7.4, SCH_2CH_3), 2.56 (2H, q, JD 7.4, SCH_2CH_3), 7.31 (2H, dd, JD 1.9, JD 7.1, Ar*H*), 8.12 (2H, dd, JD 1.9, JD 7.1, Ar*H*). ^{13}C NMR (75 MHz, $CDCl_3$) δ 13.7 (1C, SCH_2CH_3), 26.0 (1C, SCH_2CH_3), 123.9 (2C, Ar), 126.0

(2C, Ar), 144.9 (1C, Ar), 147.9 (1C, Ar). Anal. calcd. for $C_8H_9NO_2S$: C, 52.44; H, 4.95. Found: C, 52.32; H, 4.80.

1-Propylthio-4-nitrobenzene 6c ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.09 (3H, t, JD 7.3, $SCH_2CH_2CH_3$), 1.76 (2H, m, $SCH_2CH_2CH_3$), 3.01 (2H, t, JD 7.3, $SCH_2CH_2CH_3$), 7.31 (2H, dd, JD 1.8, JD 7.0, Ar*H*), 8.12 (2H, dd, JD 1.8, JD 7.0, Ar*H*). ^{13}C NMR (75 MHz, $CDCl_3$) δ 13.4 (1C, $SCH_2CH_2CH_3$), 21.8 (1C, $SCH_2CH_2CH_3$), 33.8 (1C, $SCH_2CH_2CH_3$), 123.8 (2C, Ar), 125.9 (2C, Ar), 144.7 (1C, Ar), 148.0 (1C, Ar). Anal. calcd. for $C_9H_{11}NO_2S$: C, 54.80; H, 5.62. Found: C, 54.63; H, 5.51.

1-Nitro-4-((2-propenyl)thio)-benzene 6d ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.68 (2H, t, JD 6.5, $SCH_2CH{=}CH_2$), 5.22 (1H, dd, JD 1.0, JD 10.5, $SCH_2CH{=}CH_2$), 5.33 (1H, dd, JD 1.0, JD 17.0, $SCH_2CH{=}CH_2$), 5.85 (1H, m, $SCH_2CH{=}CH_2$), 7.34 (2H, dd, JD 2.0, JD 7.0, ArH), 8.12 (2H, dd, JD 2.0, JD 7.0, ArH). ^{13}C NMR (75 MHz, $CDCl_3$) δ 35.2 (1C, $SCH_2CH{=}CH_2$), 119.0 (1C, $SCH_2CH{=}CH_2$), 123.8 (2C, Ar), 126.8 (2C, Ar), 131.9 (1C, $SCH_2CH{=}CH_2$), 145.2 (1C, Ar), 146.8 (1C, Ar). Anal. calcd. for $C_9H_9NO_2S$: C, 55.37; H, 4.65. Found: C, 55.16; H, 4.50.

1-Nitro-4-(phenylmethylthio)-benzene 6e ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.26 (2H, s, $SCH_2C_6H_5$), 7.30 (5H, m, $SCH_2C_6H_5$), 7.38 (2H, dd, JD 1.9, JD 7.5, ArH), 8.12 (2H, dd, JD 1.9, JD 7.5, ArH). ^{13}C NMR (75 MHz, $CDCl_3$) δ 37.0 (1C, $SCH_2C_6H_5$), 123.8 (2C, Ar), 126.8 (2C, Ar), 127.8 (1C, Ar) 128.7 (2C, Ar) 128.9 (2C, Ar), 135.4 (1C, Ar), 145.2 (1C, Ar), 147.3 (1C, Ar). Anal. calcd. for $C_{13}H_{11}NO_2S$: C, 63.65; H, 4.52. Found: C, 63.42; H, 4.45.

2-[(4-Nitrophenyl)thio]-1-phenyl-1-ethanone 6f ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.46 (2H, s, $SCH_2COC_6H_5$), 7.42 (2H, d, JD 8.9, ArH), 7.50 (2H, m, $SCH_2COC_6H_5$), 7.63 (1H, d, JD 7.4, $SCH_2COC_6H_5$), 8.00 (2H, d, JD 7.5, $SCH_2COC_6H_5$), 8.13 (2H, d, JD 8.9, ArH). ^{13}C NMR (75 MHz, $CDCl_3$) δ 39.1 (1C, $SCH_2COC_6H_5$), 124.0 (2C, Ar), 12H_3 (2C, Ar), 128.6 (2C, Ar) 128.9 (2C, Ar) 134.0 (1C, Ar), 134.9 (1C, Ar), 145.3 (1C, Ar), 145.6 (1C, Ar), 192.7 (1C, C=O). Anal. calcd. for $C_{14}H_{11}NO_3S$: C, 61.53; H, 4.06. Found: C, 61.22; H, 4.00.

1-Chloro-4-methylthiobenzene 6g ^{1}H NMR (300 MHz, $CDCl_3$) δ 2.47 (3H, s, SCH_3), 7.18 (2H, dd, JD 1.9, JD 8.5, ArH), 7.26 (2H, dd, JD 1.9, JD 8.5, ArH). ^{13}C NMR (75 MHz, $CDCl_3$) δ 16.0 (1C, SCH_3), 127.8 (2C, Ar), 128.9 (2C, Ar), 130.8 (1C, Ar), 137.0 (1C, Ar). Anal. calcd. for C_7H_7ClS: C, 53.00; H, 4.45. Found: C, 52.85; H, 4.39.

1-Chloro-4-ethylthiobenzene 6h ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.31 (3H, t, JD 7.4, SCH_2CH_3), 2.92 (2H, q, JD 7.4, SCH_2CH_3), 7.25 (4H, s, ArH). ^{13}C NMR (75 MHz, $CDCl_3$) δ 14.2 (1C, SCH_2CH_3), 27.9 (1C, SCH_2CH_3), 128.9 (2C, Ar), 130.3 (2C, Ar), 131.7 (1C, Ar), 135.1 (1C, Ar). Anal. calcd. for C_8H_9ClS: C, 55.65; H, 5.25. Found: C, 55.48; H, 5.14.

1-Chloro-4-propylthiobenzene 6i ^{1}H NMR (300 MHz, $CDCl_3$) δ 1.02 (3H, t, JD 7.5, $SCH_2CH_2CH_3$), 1.62 (2H, m, $SCH_2CH_2CH_3$), 2.85 (2H, t, JD 6.5, $SCH_2CH_2CH_3$), 7.22 (2H, d, JD 5.1, ArH), 7.26 (2H, d, JD 5.1, ArH). ^{13}C NMR (75 MHz, $CDCl_3$) δ 13.3 (1C, $SCH_2CH_2CH_3$), 22.4 (1C, $SCH_2CH_2CH_3$), 35.8 (1C, $SCH_2CH_2CH_3$), 128.9 (2C, Ar), 130.3 (2C, Ar), 131.6 (1C, Ar), 135.4 (1C, Ar). Anal. calcd. for $C_9H_{11}ClS$: C, 57.90; H, 5.94. Found: C, 57.77; H, 5.73.

1-Chloro-4-[(2-propenyl)thio]benzene 6j ^{1}H NMR (300 MHz, $CDCl_3$) δ 3.52 (2H, t, JD 6.9, $SCH_2CH{=}CH_2$), 5.08 (1H, dd, JD 1.4, JD 7.2, $SCH_2CH{=}CH_2$), 5.12 (1H, dd, JD 1.4, JD 14.1, $SCH_2CH{=}CH_2$) 5.85 (1H, m, $SCH_2CH{=}CH_2$), 7.26 (2H, s, ArH), 7.27 (2H, s, ArH). ^{13}C NMR (75 MHz, $CDCl_3$) δ 37.4 (1C, $SCH_2CH{=}CH_2$), 117.9 (1C, $SCH_2CH{=}CH_2$), 128.9 (2C, Ar), 131.3 (2C, Ar), 132.3 (1C, Ar), 133.2 (1C, $SCH_2CH{=}CH_2$), 134.3 (1C, Ar). Anal. calcd. for C_9H_9ClS: C, 58.53; H, 4.91. Found: C, 58.36; H, 4.78.

1-Chloro-4-(phenylmethylthio)-benzene 6k ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.11 (2H, s, $SCH_2C_6H_5$), 7.24 (4H, s, ArH), 7.31 (5H, m, $SCH_2C_6H_5$). ^{13}C NMR (75 MHz, $CDCl_3$) δ 39.2 (1C, $SCH_2C_6H_5$), 127.2 (2C, Ar), 128.5 (2C, Ar), 128.7 (2C, Ar) 128.9 (2C, Ar) 131.3 (1C, Ar), 132.4 (1C, Ar), 134.6 (1C, Ar), 137.0 (1C, Ar). Anal. calcd. for $C_{13}H_{11}ClS$: C, 66.52; H, 4.72. Found: C, 66.41; H, 4.55.

2-[(4-Chlorophenyl)thio]-1-phenyl-ethanone 6l ^{1}H NMR (300 MHz, $CDCl_3$) δ 4.25 (2H, s, $SCH_2COC_6H_5$), 7.24 (2H, dd, JD 2.0, JD 6.5, ArH), 7.31 (2H, dd, JD 2.0, JD 6.5, ArH), 7.48 (2H, m, $SCH_2COC_6H_5$), 7.58 (1H, d, JD 7.4, $SCH_2COC_6H_5$), 7.94 (2H, d, JD 8.6, $SCH_2COC_6H_5$). ^{13}C NMR (75 MHz, $CDCl_3$) δ 41.2 (1C, $SCH_2COC_6H_5$), 128.6 (2C, Ar), 128.7 (2C, Ar), 129.2 (2C, Ar) 131.9 (2C, Ar) 133.1 (1C, Ar), 133.3 (1C, Ar), 133.6 (1C, Ar), 135.2 (1C, Ar), 193.7 (1C, C=O). Anal. calcd. for $C_{14}H_{11}ClOS$: C, 64.00; H, 4.22. Found: C, 63.85; H, 4.11.

REFERENCES

[1] For general reviews on sulfides, see *Comprehensive Organic Chemistry*, D. N. Jones, Ed., Vol. 3, pp. 33–103 (Pergamon Press, Oxford, 1979).

[2] P. S. Herradura, K. A. Pendola, and R. K. Guy, *Org. Lett.*, **2,** 2019 (2000), and references therein.

[3] (a) J. Lindley, *Tetrahedron*, **40,** 1433 (1984); (b) S.-I. Yunoki, K. Takimiya, Y. Aso, and T. Otsubo, *Tetrahedron Lett.*, **38,** 3017 (1997); (c) J. Yin and C. Pidgeon, *Tetrahedron Lett.*, **38,** 5953 (1997).

[4] S. T. A. Shah, K. M. Khan, A. M. Heinrich, and W. Voelter, *Tetrahedron Letters*, **43,** 8281 (2002).

[5] (a) C. Goux, P. Lhoste, and D. Sinou, *Tetrahedron Lett.*, **33,** 8099 (1992); (b) P. C. B. Page, S. S. Klair, M. P. Brown, M. M. Harding, C. S. Smith, S. J. Maginn, and S. Mulley, *Tetrahedron Lett.*, **29,** 4477 (1988); (c) C.-J. Li and D. N. Harpp, *Tetrahedron Lett.*, **33,** 7293 (1992).

[6] S. Chowdhury and S. Roy, *Tetrahedron Letters*, **38,** 2149 (1997).
[7] (a) F. Y. Kwong and S. L. Buchwald, *Org. Lett.*, **4,** 3517 (2002); (b) C. G. Bates, R. K. Gujadhur, and D. Venkataraman, *Org. Lett.*, **4,** 2803 (2002); (c) N. Taniguchi and T. Onami, *J. Org. Chem.*, **69,** 915 (2004).
[8] B. C. Ranu and T. Mandal, *J. Org. Chem.*, **69,** 5793 (2004).
[9] For reviews on the application of sodium dithionite in organic synthesis, see (a) J. Cho, S. Sato, S. Takeoka, and E. Tsuchida, *Macromolecules*, **34,** 2751 (2001); (b) C. G. Overberger, E. V. McGill, and J. P. Anselme, *J. Am. Chem. Soc.*, **91,** 687 (1969); (c) J. O. Hawthorne, E. L. IMihelic, M. S. Morgan, and M. H. Wilt, *J . Org. Chem.*, **28,** 2831 (1963); (d) U. Eisner and J. Kuthan, *Chem. Rev.*, **72,** 1 (1972); (e) J. P. Ferris, U. S. Pat. 3670007 (1972); (f) L. I. Smith and W. M. Schubert, *J. Am. Chem. Soc.*, **70**, 2656 (1948); (g) M. J. Haddadin, H. N. Alkaysi, and S. E. Saheb, *Tetrahedron*, **26**, 1115 (1970); (h) E. D. Basalkevich, T. I. Cherepenko, N. N. Vysatskii, V. F. Shapo-valenko, and A. A. Svishchuk, *Fiziol. Akt. Veshchestua*, 174 (1971); *Chem. Abstr.*, **77,** 61449 (1972); (i) M. Furusawa, S. Iwastdti, and Y. Matsumura, *Nippon Kagaku Kaishi*, 2228 (1974); *Chem. Abstr.*, **82,** 38271 (1975); (j) M. A. Brimble and M. R. Nairn. *J. Chem. Soc.*, *Perkin Trans. 1*, 317 (2000); (k) J. G. de Vries and R. M. Kellogg, *J . Org. Chem.*, **45,** 4126 (1980); (l) P. Cao, J. X. Duan, and Q. Y. Chen, *J. Chem. Soc. Chem. Commun.*, 737 (1994); (m) F. H. Wu and B. N. Huang, *Chin. Chem . Lett.*, **4,** 308 (1993); (n) W. Y. Huang and Y. M. Wu, Chinese Pat. CN 91 107446 (1991); (o) W. Y. Huang, L. Q. Hu, and W. Z. Ge, *J. Fluorine Chem.*, **43,** 305 (1989); (p) X. Q. Tang and C. M. Hu, *J. Chem. Soc. Chem. Commun.*, 631 (1994); (q) M. Tordeux, B. Langlois, and C. Wakselman, *J. Chem. Soc. Perkin Trans. 1*, 2293 (1990).
[10] R. Y. Tang, P. Zhong, and Q. L. Lin, *Journal of Fluorine Chemistry*, **127,** 948 (2006).
[11] W. G. Hcdgson, A. Neaves, and C. A. Parker, *Nature (London)*, **178,** 489 (1956).
[12] S. Antonello, K. Daasbjerg, H. Jensen, F. Taddei, and F. Maran, *J. Am. Chem. Soc.*, **125,** 14905 (2003).
[13] S. Antonello, R. Benassi, G. Gavioli, F. Taddei, and F. Maran, *J. Am. Chem. Soc.*, **124,** 7529 (2002).
[14] M. A. Aubart and R. G. Bergman, *J. Am. Chem. Soc.*, **120,** 8755 (1998).
[15] R. Benassi, G. L. Fiandri, and F. Taddei, *Journal of Molecular Structure (Themhem)*, **418,** 127 (1997).
[16] T. Nishino, M. Okada, T. Kuroki, T. Watanabe, Y. Nishiyama, and N. Sonoda, *J. Org. Chem.*, **67,** 8696 (2002).