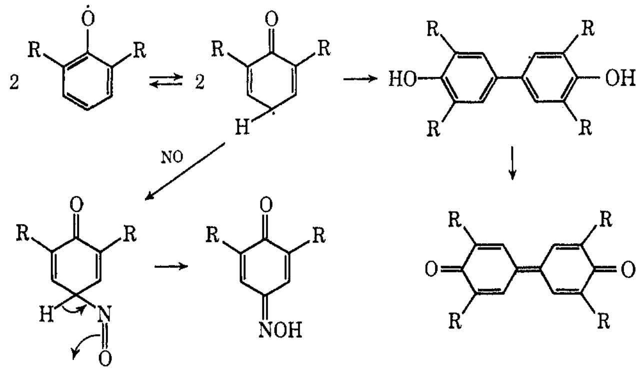
The reduction in yield when a *t*-butyl group is present is probably due to two factors: the increased solubility of diphenoquinones containing such groups and competing oximination. The isolation of 2,6-di-*t*-butyl-4-oximinobenzoquinone, which has been reported both as *p*-nitroso-2,6-di-*t*-butylphenol[4] or as the oxime,[5] and recently has been shown to exist as the oxime,[6] from the oxidation of 2,6-di-*t*-butyl phenol supports the second factor.

The reaction path very likely involves the production of phenoxy radicals. Ershov and Zlobina were able to record the esr signal for 2,4,6-trisubstituted phenoxy radicals when the corresponding phenols were oxidized with alkyl nitrites.[2b] They proposed that an alkoxy radical, produced by scission of the alkyl nitrite, abstracted hydrogen from the phenol to give the phenoxy radical. Some support for the intermediacy of an alkoxy radical is contained in the thermal decomposition of 2-octyl nitrite at 100°.[7] The products are the 2-octyloxy and the nitric oxide radicals. In the case of the 2,6-disubstituted phenols in Table I, the free *para* positions would allow dimerization to the biphenol.[8] Subsequent oxidation of the biphenol would give the diphenoquinone, Scheme I. The total process involves the removal of two hydrogen atoms from each mole of phenol to give the diphenoquinone, necessitating at least 2 mol of oxidant/mol of phenol. The formation of oxime in the case of 2,6-di-*t*-butylphenol can be explained by the combination of nitric oxide and phenoxy radicals (Scheme I).

Scheme I



Experimental Section[9]

3,3′,5,5′-Tetramethyl-4,4′-diphenoquinone.—An example of the general method is given for 2,6-dimethylphenol. 2,6-Dimethylphenol (1.83 g, 0.015 mol) was dissolved in 50 ml of methylene chloride and isoamyl nitrite added (4.3 ml, 0.032 mol). After stirring at ambient temperature for 24 hr, the reaction was worked up by cooling and filtering. The red crystals obtained were washed with several portions of cold methylene chloride and dried to give 0.976 g of product (53%), mp 205.5–208° (lit.[10] mp 207–210°). The ir spectrum was essentially identical with that of the authentic material.

3,3′,5,5′-Tetra-*t*-butyl-4,4′-diphenoquinone.—2,6-Di-*t*-butylphenol (3.09 g, 0.015 mol) and isoamyl nitrite (6.72 ml, 0.050 mol) were dissolved in 20 ml of methylene chloride. The solution was stirred at ambient temperature for 28 hr and then heated at reflux for 15 hr. The solvent was distilled to leave a semisolid mass. This was treated with acetic acid and filtered. The filter cake was washed with acetic acid and dried to give 0.503 g of title compound (16.3%), mp 242.5–244° (lit.[11] mp 245–247°). The ir spectrum was essentially identical with that of the authentic material.

2,6-Di-*t*-butyl-4-oximino-*p*-benzoquinone.—2,6-Di-*t*-butylphenol (3.09 g, 0.015 mol) was dissolved in 25 ml of methylene chloride and isoamyl nitrite (5.10 ml, 0.0375 mol) was added. The reaction solution was stirred at ambient temperature for 27 hr and then the solvent was removed under vacuum (20 mm). The gummy dark residue was treated with 20 ml of methyl alcohol and filtered to remove some brown solid. The filtrate was warmed under vacuum to remove the solvent and the dark oil which remained was extracted with hot hexane. The hexane was concentrated and cooled to give 0.273 g of yellow crystals, mp 205–207°. Further hexane extraction of the dark gum gave an additional 0.370 g of yellow crystals, mp 199–205°. The two batches were combined and recrystallized from hexane to give 0.630 g (18%) of title compound, mp 213–214° (lit.[5] mp 219–220°). The ir spectrum contained a broad band at 3320 cm^{-1} (-OH) and a sharp band at 1600 cm^{-1} (C=N).

3,3′-Dichloro-5,5′-diphenyl-4,4′-diphenoquinone.—2-Chloro-6-phenylphenol (2.04 g, 0.01 mol) was dissolved in 22 ml of methylene chloride and isoamyl nitrite was added (4.0 ml, 0.03 mol). The solution was stirred at ambient temperature for 24 hr at which time some red solid was present. The mixture was heated at reflux for 2 hr, cooled, and filtered. The filter cake was washed with cold methylene chloride and dried under vacuum to give purple-red crystals, 0.21 g (10%), mp 282–283°. This was recrystallized from chloroform to give mp 287.5–288.5°; ir (KBr) 1610 cm^{-1} (C=O). *Anal.* Calcd for $C_{24}H_{14}Cl_2O_2$: C, 71.13; H, 3.48; Cl, 17.50. Found: C, 70.5; H, 3.77; Cl, 17.7.

Registry No.—Isoamyl nitrite, 110-46-3; 2,6-dimethylphenol, 576-26-1; 2,6-diphenylphenol, 2432-11-3; 2-methyl-6-phenylphenol, 17755-10-1; 2,6-dimethoxyphenol, 91-10-1; 2,6-di-*t*-butylphenol, 128-39-2; 2-chloro-6-phenylphenol, 85-97-2; 2,6-dichlorophenol, 87-65-0; 3,3′-dichloro-5,5′-diphenyl-4,4′-diphenoquinone, 24378-09-4.

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β-Keto Sulfoxides. IX. Conversion into Acetylenic Sulfoxides and Sulfones[1]

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We have previously described the conversion of esters to β-keto sulfoxides (reaction 1)[2] and the reduction of the β-keto sulfoxide to the hydroxy sulfide (reaction 2),[3] which may be dehydrated to give the vinyl sulfide (reaction 3).[3] Attempts to convert β-(methylmercapto)styrene to the acetylenic sulfide by the standard bromination–dehydrobromination technique[4]

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(1) Part VIII: G. A. Russell and L. A. Ochrymowycz, *J. Org. Chem.*, **35**, 764 (1970). This work was supported by a grant from the U. S. Army Research Office (Durham).

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gave in our hands only a 13% yield of methyl phenethynyl sulfide. We have therefore developed an alternate method (reactions 4–6) for converting a vinyl sulfide into an acetylenic sulfide using the previously described reactions 5 and 6.[3]

$$RCO_2Et + CH_3SOCH_2^- \longrightarrow RCOCH_2SOCH_3 + EtO^- \quad (1)$$

$$RCOCH_2SOCH_3 \xrightarrow{LiAlH_4} RCH(OH)CH_2SCH_3 \quad (2)$$

$$RCH(OH)CH_2SCH_3 \xrightarrow{H^+, \Delta} RCH{=}CHSCH_3 \quad (3)$$

$$RCH{=}CHSCH_3 \xrightarrow{NaIO_4} RCH{=}CHSOCH_3 \quad (4)$$

$$RCH{=}CHSOCH_3 \xrightarrow{SOCl_2} RC(Cl){=}CHSCH_3 \quad (5)$$

$$RC(Cl){=}CHSCH_3 \xrightarrow[EtOH]{KOH} RC{\equiv}CSCH_3 \quad (6)$$

$$RC{\equiv}CSCH_3 \xrightarrow{m\text{-}ClC_6H_4CO_3H} RC{\equiv}CSOCH_3 \xrightarrow{[O]} RC{\equiv}CSO_2CH_3 \quad (7)$$

The oxidation of vinyl sulfides to vinyl sulfoxides is complicated by epoxidation of the double bond and the possibility of overoxidation to yield the sulfone. We have found that a selective oxidation of a vinyl sulfide to a vinyl sulfoxide can be achieved using sodium metaperiodate in a modification of the procedure of Leonard and Johnson.[5] Nearly quantitative yields of vinyl sulfoxides can be obtained by the use of 0.5 *M* sodium metaperiodate in 50% aqueous acetonitrile solution at −10°. An alternate procedure, which is greatly preferred for the conversion of acetylenic sulfides to acetylenic sulfoxides, is to use 1 equiv of *m*-chloroperbenzoic acid at −20° in chloroform solution.[6] Table I summarizes some pertinent results.

TABLE I

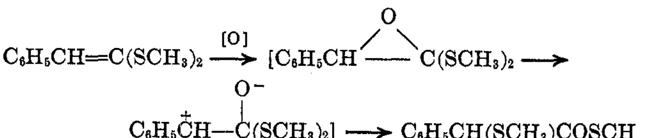
CONVERSION OF SULFIDES TO SULFOXIDES

Sulfide	—Yield (%) of sulfoxide— NaIO₄	*m*-ClC₆H₄CO₃H
$C_6H_5CH{=}CHSCH_3$	77[a]	
$C_6H_5C{\equiv}CSCH_3$	46	92
$C_6H_5CH{=}C(CH_3)SCH_3$	74	
$C_6H_5C(Cl){=}CHSCH_3$	73	
$(C_6H_5)_2C{=}CHSCH_3$	96	79
$(C_6H_5)_2C{=}C(SCH_3)_2$	96	97
$C_6H_5C(CH_3){=}C(SCH_3)_2$	90	90
$C_6H_5C(C_2H_5){=}C(SCH_3)_2$	68	86

[a] See ref 3.

m-Chloroperbenzoic acid (2 equiv) is the preferred reagent to convert methyl phenethynyl sulfide to the sulfone (81% yield). Use of hydrogen peroxide in acetic acid[4,7] led only to a polymer under the conditions reported to be satisfactory for the oxidation of phenyl phenethynyl sulfide.

β,β-Di(methylmercapto)styrene reacted with either sodium metaperiodate or *m*-chloroperbenzoic acid to yield a rearrangement product [S-methyl α-(methylmercapto)phenylthioacetic acid] probably the result of epoxidation of the double bond.[1]



An alternate reaction sequence to reactions 6 and 7 is to first oxidize the chloro vinyl sulfide to the sulfoxide and then dehydrohalogenate (reactions 8 and 9).

$$C_6H_5C(Cl){=}CHSCH_3 \xrightarrow{[O]} C_6H_5C(Cl){=}CHSOCH_3 \quad (8)$$

$$C_6H_5C(Cl){=}CHSOCH_3 \xrightarrow[THF]{KOC(CH_3)_3} \underset{(89\%)}{C_6H_5C{\equiv}CSOCH_3} \quad (9)$$

$$C_6H_5C(Cl){=}CHSOCH_3 \xrightarrow[THF]{NaOC_2H_5} \underset{(86\%)}{C_6H_5C(OC_2H_5){=}CHSOCH_3} \quad (10)$$

Use of potassium *t*-butoxide in THF, a base with low nucleophilicity, led to methyl phenethynyl sulfoxide in high yield, whereas a nucleophilic base, such as sodium ethoxide, led to the α-ethoxy-β-(methylsulfinyl)styrene. Ethoxide ion is known to add readily to acetylenic sulfides[8] and will presumably add even more readily to acetylenic sulfoxides.

Experimental Section[9]

Oxidation Techniques.—The sodium metaperiodate oxidations were performed by mixing at −10° 60 ml of a acetonitrile solution containing 25 mmol of the sulfide with 60 ml of a 0.5 *M* aqueous solution of sodium metaperiodate. The solution was stirred in a refrigerator at −5 to −10° for 12 hr, whence the cold solution was filtered and extracted three times with 50-ml portions of chloroform. The dry chloroform extracts ($MgSO_4$) were concentrated under vacuum to yield the crude sulfoxides that were purified by column chromatography or crystallization.

For the *m*-chloroperbenzoic acid oxidations, the vinyl sulfide (25 mmol) in 100 ml of chloroform was cooled to −10° and 1 equiv of analyzed *m*-chloroperbenzoic acid[10] in 50 ml of chloroform at −10° was added slowly. The reaction flask was stoppered and allowed to stand at −23° in a refrigerator for 12 hr. The cool solution was filtered to remove *m*-chlorobenzoic acid, washed twice with 100 ml of saturated aqueous sodium bicarbonate, and dried ($MgSO_4$); the solvent was removed under vacuum to yield the crude sulfoxide.

Sulfoxides.—The vinyl sulfides listed in Table I have been previously described,[1,3] as has **β-(methylsulfinyl)styrene.**[3] **1-(Methylsulfinyl)-2,2-diphenylethylene** was crystallized from 1:1 hexane–ether to yield material: mp 106°; pmr δ 2.72 (s, 3, $SOCH_3$), 6.81 (s, 1, =CH–), δ 7.15–7.55 (m, 10, C_6H_5).

Anal. Calcd for $C_{15}H_{14}OS$: C, 74.36; H, 5.83; S, 13.21. Found: C, 74.10; H, 5.92; S, 13.21.

β-Methyl-β-(methylsulfinyl)styrene was crystallized from 1:4 ethyl acetate–hexane to give mp 103–104°; pmr δ 2.63 (s, 3, $SOCH_3$), 2.21 (d, 3, CH_3, $J = 2$ Hz), 7.10 (q, 1, =CH–, $J = 2$ Hz), 7.32 (s, 5, C_6H_5).

Anal. Calcd for $C_{10}H_{12}OS$: C, 66.65; H, 6.71; S, 17.76. Found: C, 66.75; H, 6.60; S, 17.93.

α-Chloro-β-(methylsulfinyl)styrene was purified by column chromatography on silica gel. Impurities were eluted by ethyl acetate and the sulfoxide was recovered by elution with methanol. Evaporation of the methanol gave a product that was recrystallized from 1:1 ethyl acetate–hexane to give the sulfoxide: mp 85–86°; pmr δ 2.80 (s, 3, $SOCH_3$), 6.98 (s, 1, =CH–), 7.30–7.80 (m, 5, C_6H_5).

Anal. Calcd for C_9H_9ClOS: C, 53.86; H, 4.52; S, 15.97; Cl, 17.66. Found: C, 54.01; H, 4.50; S, 16.02; Cl, 17.74.

α-Methyl-β-(methylsulfinyl)styrene was prepared from a mixture of 9 parts of *cis-* to 1 part of *trans*-α-methyl-β-(methylmercapto)styrene[3] (*cis* and *trans* refer to the relationship of the phenyl and thiomethyl groups). The crude product appeared

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(9) Pmr spectra were obtained in $CDCl_3$ at 60 MHz. Mass spectra were obtained by direct inlet into an Atlas Werke CH-4 spectrometer.

(10) Material from the Research Organic/Inorganic Chemical Co., Sun Valley, Calif., was used in this work; assay, 83% peracid.

to be a mixture of the *cis* and *trans* sulfoxides in ~9:1 ratio. Column chromatography on silica gel with ethyl acetate eluent gave material that could be crystallized from 4:1 hexane–ethyl acetate to yield material with mp 28–34°. Pmr: predominant isomer (90%), δ 2.68 (s, 3, $SOCH_3$), 2.39 (d, 3, CH_3, J = 1.5 Hz), 6.67 (q, 1, =CH–, J = 1.5 Hz); minor isomer (10%), δ 2.61 (s, 3, $SOCH_3$), 2.28 (d, 3, CH_3, J = 2.5 Hz), 6.40 (q, 1, =CH–, J = 2.5 Hz).

Anal. Calcd for $C_{10}H_{12}OS$: C, 66.65; H, 6.71; S, 17.76. Found: C, 66.83; H, 6.76; S, 17.88.

α-Ethyl-β-(methylsulfinyl)styrene was prepared from a mixture of 8 parts of *cis*- and 2 parts of *trans*-α-ethyl-β-(methylmercapto)-styrene[3] (*cis* and *trans* refer to the relationship of the phenyl and thiomethyl groups). The nearly pure crude product was chromatographed from silica gel by ethyl acetate to remove traces of more and less mobile impurities to give an oil which contained approximately 80% *cis* sulfoxide and 20% *trans* sulfoxide. Pmr: predominant isomer (80%), δ 2.67 (s, 3, $SOCH_3$), 6.46 (s, 1, =CH–); minor isomer (20%), δ 2.58 (s, 3, $SOCH_3$); 6.32 (t, 1, =CH–, J = 2 Hz). The ethyl groups and aryl groups gave pmr multiplets centered at δ 1.07, 2.85, and 7.3.

Anal. Calcd for $C_{11}H_{14}OS$: C, 68.02; H, 7.27; S, 16.47. Found: C, 67.99; H, 7.43; S, 16.32.

1-(Methylsulfinyl)-1-(methylmercapto)-2,2-diphenylethylene was recrystallized from 1:1 ethyl acetate–hexane to give material: mp 108–110°; pmr δ 2.24 (s, 3, SCH_3), 2.73 (s, 3, $COCH_3$), 7.10–7.45 (broad s, 10, C_6H_5).

Anal. Calcd for $C_{16}H_{16}OS_2$: C, 66.66; H, 5.59; S, 22.20. Found: C, 66.39; H, 5.41; S, 22.49.

β-(Methylsulfinyl)-β-(methylmercapto)-α-methylstyrene was recrystallized from 4:1 hexane–ether to give material, mp 101–102°, apparently a single stereoisomer: pmr δ 2.15 (s, 3, CH_3), 2.40 (s, 3, SCH_3), 2.62 (s, 3, $SOCH_3$), 7.00–7.45 (m, 5, C_6H_5).

Anal. Calcd for $C_{11}H_{14}OS_2$: C, 58.40; H, 6.24; S, 28.29. Found: C, 58.56; H, 6.02; S, 28.30.

β-(Methylsulfinyl)-β-(methylmercapto)-α-ethylstyrene was crystallized from 4:1 hexane–ether to give crystals, mp 82–84°, apparently a single stereoisomer: pmr δ 0.99 (t, 3, CH_2CH_3, J = 7.5 Hz), 2.58 (broad s, 6, SCH_3, $SOCH_3$, Δδ = 0.8 Hz), 2.94 (q, 2, CH_2CH_3, J = 7.5 Hz), 6.95–7.45 (m, 5, C_6H_5).

Anal. Calcd for $C_{12}H_{16}OS_2$: C, 59.99; H, 6.71; S, 26.64. Found: C, 60.00; H, 6.62; S, 26.62.

α-Chloro-β-(methylsulfinyl)styrene[3] **(Reaction 5)**.—α-(Methylmercapto)styrene (20 g, 120 mmol) in 100 ml of methylene chloride was treated with 18 ml (250 mmol) of thionyl chloride dissolved in 20 ml of methylene chloride. The thionyl chloride solution was added dropwise at a rate sufficient to maintain reflex. After addition of the thionyl chloride the mixture was stirred for 4 hr at 25°. Removal of the solvent under vacuum left a brown oil which was dissolved in 300 ml of hexane and washed twice with 100 ml of 10% $NaHCO_3$ and dried over $MgSO_4$. Distillation yielded 17.5 g (79%) of the chloro sulfide: bp 76–82° (0.2 Torr); pmr ($CDCl_3$) δ 2.45 (s, 3, SCH_3); 6.60 (s, 1, –CH=), 7.18–7.55 (m, 5, C_6H_5); ir (neat) 1670 cm^{-1} (C=C).

Methyl Phenethynyl Sulfide (Reaction 6).—α-Chloro-β-(methylsulfinyl)styrene (39 g, 0.21 mol) in 150 ml of ethanol was added dropwise to a mixture of 29 g of KOH (0.5 mol) in 100 ml of ethanol. The mixture was refluxed overnight and filtered, and the solvent was evaporated by use of a water aspirator. The residue was distilled to give 23 g (63%) of material boiling at 85–87° (2 Torr), lit.[11] bp 74° (2 Torr).

Reactions 5 and 6 can be combined into a single operation. Thus, the crude chloro sulfide obtained from 175 mmol of ω-(methylmerapto)styrene upon removal of the methylene chloride was dissolved in 50 ml of ethanol and added to a mixture of 25 g of KOH in 200 ml of ethanol. After stirring for 12 hr at 60° the reaction was filtered and concentrated under vacuum. Dilution with 100 ml of H_2O was followed by extraction twice with 200-ml portions of *n*-hexane and drying over $MgSO_4$; distillation yielded 17 g (67%) of methyl phenethynyl sulfide.

Methyl Phenethynyl Sulfoxide (Reaction 7).—The sulfoxide was best prepared by oxidation of the sulfide with *m*-chloroperbenzoic acid as described earlier. The sulfoxide was isolated by removal of the chloroform solvent at room temperature under vacuum. The product was eluted from a 2.5 × 50 cm silica gel column by 1.5:1 ethyl acetate–hexane as a colorless oil which polymerized upon standing: ir (CCl_4) 1060 (S=O), 2210 (C≡C) cm^{-1}; pmr (60 MHz, $CDCl_3$) δ 2.98 (s, 3, $SOCH_3$), 7.2–7.6 (m, 5, C_6H_5).

Anal. Calcd for C_9H_8OS: C, 65.58; H, 4.91; S, 19.49. Found: C, 65.70; H, 5.03; S, 19.54.

Methyl Phenethynyl Sulfone.—Methyl phenethynyl sulfide (2.96 g, 20 mmol) was dissolved in 100 ml of chloroform and cooled to −10°. A solution of 7.9 g of 83% *m*-chloroperbenzoic acid in 100 ml of chloroform at −10° was added slowly to the sulfide, and the mixture was allowed to stand for 4 days at −20°. The *m*-chlorobenzoic acid was filtered from the cool solution, and the chloroform solutions were washed twice with 100 ml of saturated sodium bicarbonate and dried ($MgSO_4$); the solvent was removed under vacuum. The residue was crystallized from 2:1:7 ethyl acetate–ether–hexane to yield 2.88 g (81%) of crystals: mp 59–60° (recrystallization from 1:1 ether–hexane raised this to 61–62°); ir (KBr) 1308 (SO_2), 1145 (SO_2), 1125 (SO_2), 2180 (C≡C) cm^{-1}; pmr δ 3.30 (s, 3, SO_2CH_3); 7.25–7.75 (m, 5, C_6H_5).

Anal. Calcd for $C_9H_8O_2S$: C, 60.00; H, 4.48; S, 17.77. Found: C, 60.11; H, 4.48; S, 17.58.

Dehydrochlorination of α-Chloro-β-(methylsulfinyl)styrene (Reaction 9).—Reaction of 2.0 g (10 mmol) of the chloro sulfoxide with 2.3 g (200 mmol) of potassium *t*-butoxide in 100 ml of THF for 15 hr at 25° led to 1.31 g (89.3%) of methyl phenethynyl sulfoxide. The acetylenic sulfoxide was isolated by pouring the reaction mixture into 200 ml of ice–water followed by extraction with three 75-ml portions of chloroform. The chloroform extracts were washed with water and dried ($MgSO_4$) and the solvent was removed under vacuum.

α-Ethoxy-β-(methylsulfinyl)styrene (Reaction 10).—Treatment of 2.00 g (10 mmol) of α-chloro-β-(methylsulfinyl)styrene in 100 ml of THF with 200 mmol of sodium ethoxide yielded a crude oil that was chromatographed to yield 1.80 g (86%) of α-ethoxy-β-(methylsulfinyl)styrene: pmr δ 2.70 (s, 3, $SOCH_3$), 1.30 (t, 3, CH_2CH_3, J = 8 Hz), 3.97 (q, 2, CH_2CH_3, J = 8 Hz), 6.02 (s, 1, =CH–), 7.30–7.65 (m, 5, C_6H_5).

Anal. Calcd for $C_{11}H_{14}O_2S$: C, 62.84; H, 6.71; S, 15.22. Found: C, 62.62; H, 6.86; S, 15.13.

Registry No.—1-(Methylsulfinyl)-2,2-diphenylethylene, 21147-11-5; β-methyl-β-(methylsulfinyl)styrene, 24378-01-6; α-chloro-β-(methylsulfinyl)styrene, 24377-96-6; α-methyl-β-(methyl-sulfinyl)styrene (*cis*), 24377-97-7; α-methyl-β-(methylsulfinyl)styrene (*trans*), 24377-98-8; α-ethyl-β-(methylsulfinyl)styrene, (*cis*), 24377-99-9; α-ethyl-β-(methylsulfinyl)styrene (*trans*), 24378-00-5; 1-(methylsulfinyl)-1-(methylmercapto)-2,2-diphenylethylene, 24407-42-9; β-(methylsulfinyl)-β-(methylmercapto)-α-methylstyrene, 24378-02-7; β-(methylsulfinyl)-β-(methylmercapto)-α-ethylstyrene, 24378-03-8; methyl phenethynyl sulfoxide, 24378-04-9; methyl phenethynyl sulfone, 24378-05-0; α-ethoxy-β-(methylsulfinyl)styrene, 24378-06-1.

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