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Oxidation of Polyfunctional Sulfides with Chlorine Dioxide

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Abstract—3-Benzylsulfanyl-4,5-diphenyl-4*H*-1,2,4-triazole, 5-methylsulfanyl-1-phenyl-1*H*-tetrazole, 2-methylsulfanyl-1*H*-benzimidazole, 2-benzylsulfanyl-1*H*-benzimidazole, and 1-butylsulfanyl-4-nitrobenzene were oxidized to the corresponding sulfoxides with chlorine dioxide using different modes of oxidant supply. The oxidation process was characterized by high chemoselectivity.

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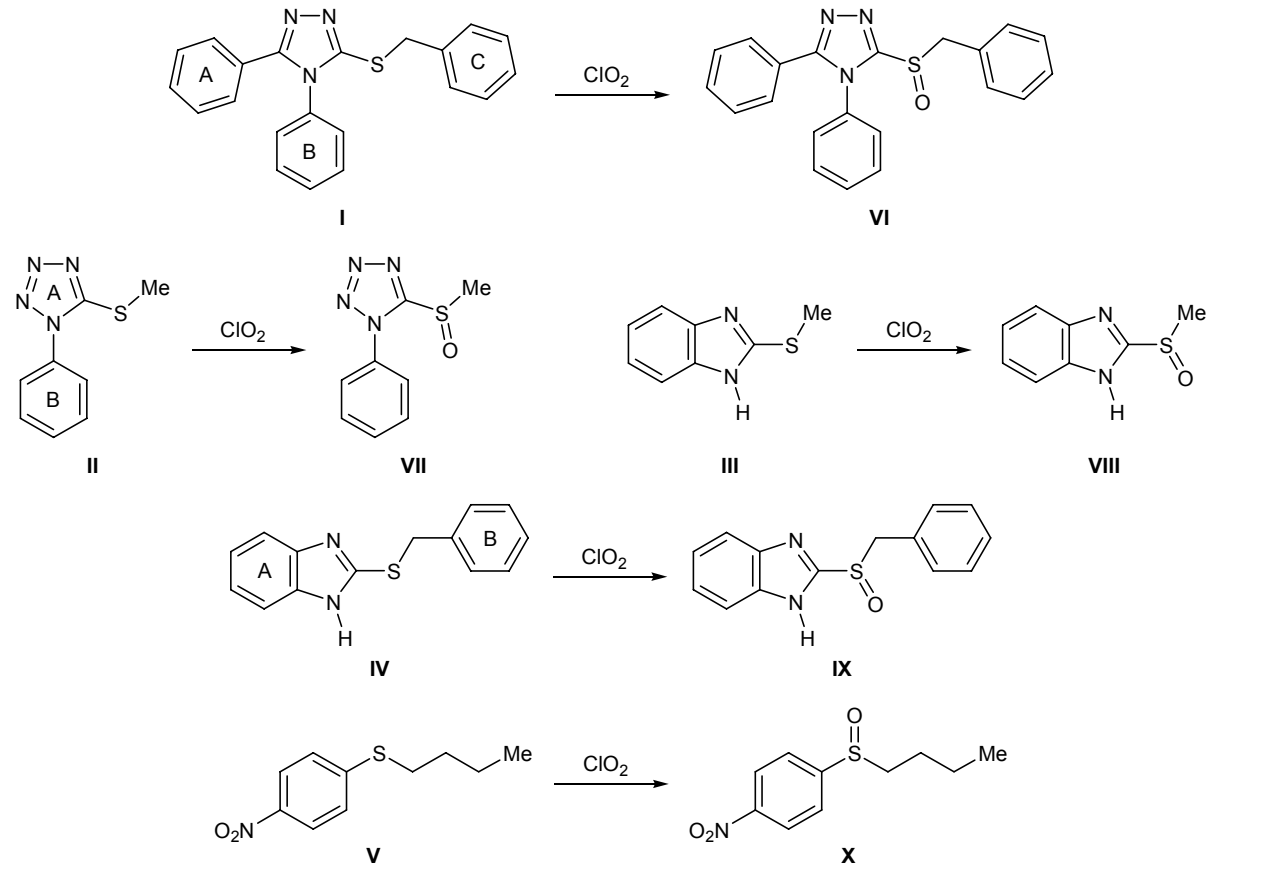
Polyfunctional sulfoxides possess important practical properties and attract increased interest. Broad spectrum of applications of polyfunctional sulfoxides is determined by their high reactivity originating from their simultaneously hydrophilic and lipophilic properties, as well as from the ability to participate in membrane processes. Polyfunctionalized sulfoxides are used as antioxidants, antidepressants [1], and stomach acid inhibitors [2, 3]. The use of sulfoxides in organic synthesis is based on their ability to promote stereo- and regioselective formation of new bonds in organic compounds [4–6]. The synthesis of polyfunctional sulfoxides and sulfones by oxidation of sulfides with different oxidants was reported in [7–15]. However, these reactions were not always chemoselective, and in some cases the formation of sulfones was difficult to control. The necessity of oxidizing polyfunctional sulfides in both aqueous and nonaqueous media stimulates search for new oxidants.

In the present article we report on the results of oxidation of polyfunctional sulfides to the corresponding sulfoxides with chlorine dioxide using different modes of oxidant supply. The use of ClO_2 in the oxidation of organic sulfides was described by us previously [16–19], and chlorine dioxide was shown to be a convenient and selective oxidant. Initial 3-benzylsulfanyl-4,5-diphenyl-4*H*-1,2,4-triazole (**I**) was synthesized according to the procedure reported in [20], and compounds **II**–**V** were provided by *Vekton* closed corporation. The structure of sulfide **I** was proved by X-ray analysis (Fig. 1). According to the X-ray diffraction data, molecule **I** is characterized by bond lengths and bond angles typical of structurally related compounds. The phenyl substituent on N^4 is turned through a dihedral angle of 81.1(2)° with respect to the heteroring plane, and the substituent on C^5 with the 1,2,4-triazole ring plane forms a dihedral angle of 34.7(2)°.

The oxidation of sulfides **I**–**V** with an equimolar amount of chlorine dioxide at 20°C gave 85–98% of the corresponding sulfoxides **VI**–**X** which contained no sulfone impurity (Scheme 1). Different oxidation conditions were applied: (*a*) oxidation with aqueous chlorine dioxide, (*b*) oxidation with a solution of ClO_2 in an organic solvent, and (*c*) bubbling of gaseous ClO_2 through the substrate solution. We previously compared [17] different oxidation procedures and found that the mode of oxidant supply almost does not affect the yield. Analogous results were obtained in the present work. After chromatographic separation in a column charged with silica gel, the yields of sulfoxides **VI**–**X** were 85–97%.

The structure of compounds **VI**–**X** was confirmed by IR and NMR spectroscopy. The IR spectra of **VI**–**X** contained absorption bands in the region 1020–1050 cm^{-1} due to stretching vibrations of the sulfoxide group. In the ^{13}C NMR spectra, signals from carbon

Scheme 1.



atoms linked to sulfur (methylene carbon atoms in **VI**, **IX**, and **X** and methyl carbon atoms in **VII** and **VIII**) were displaced downfield relative to the corresponding signals of initial sulfides. Compound **VI** displayed in the ^{13}C and ^{1}H NMR spectra signals from aromatic rings, indicating conservation of the molecular skeleton. The formation of sulfoxide group followed from the downfield shift of the $S(O)CH_2$ signal in the ^{13}C NMR spectrum (δ_C 58.82 ppm) and the presence of two doublets at δ 4.72 and 5.05 ppm from the corresponding methylene protons in the ^{1}H NMR spectrum.

The structure of compound **VII** was unambiguously determined by NMR spectroscopy and X-ray analysis. In the ^{13}C NMR spectrum of **VII**, as well as in the spectrum of initial sulfide **II**, signals from the phenyl and methyl carbon atoms were observed. The CH_3 signal appeared as δ_C 35.78 ppm. Aromatic protons gave two multiplets at δ 7.55–7.72 and 7.88–7.95 ppm in the ^{1}H NMR spectrum, and protons in the methyl group resonated as a singlet at δ 2.41 ppm.

Figure 2 shows the structure of molecule **VII** according to the X-ray diffraction data. Compound **VII** crystallizes in $P2_1/c$ centrosymmetric space group belonging to monoclinic crystal system. The benzene ring is turned through a dihedral angle of 49.8(2)° with respect to the tetrazole ring plane. The methyl group lies in the tetrazole ring plane [deviation of C^7 from the mean-square tetrazole plane is 0.173(2) Å], and the S–O bond length is 1.479(2) Å. The mode of crystal

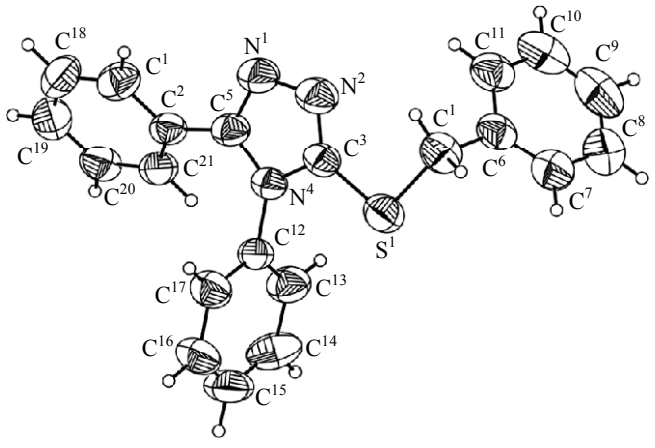


Fig. 1. Structure of the molecule of 3-benzylsulfanyl-4,5-diphenyl-4*H*-1,2,4-triazole (**I**) according to the X-ray diffraction data. Non-hydrogen atoms are shown as thermal vibration ellipsoids with a probability of 50%.

Table 1. Principal crystallographic parameters of compounds **I** and **VII**–**IX** and parameters of X-ray diffraction experiments

Parameter	**I**	**VII**	**VIII**	**IX**
Formula	$C_{21}H_{17}N_3S$	$C_8H_8N_4OS$	$C_8H_8N_2OS$	$C_{14}H_{12}N_2OS$
M	343.44	208.24	180.22	256.32
Temperature, K	295(2)	295(2)	295(2)	120(2)
Crystal system	Triclinic	Monoclinic	Monoclinic	Hexagonal
Space group	*P*1	$P2_1/c$	$P2_1$	$P6_2$
Unit cell parameters:				
a, Å	9.4769(15)	14.7007(6)	9.970(2)	13.1079(4)
b, Å	9.5228(16)	4.3831(2)	4.7081(13)	13.1079(4)
c, Å	10.0307(17)	16.4015(5)	18.667(5)	12.4005(6)
α, deg	102.297(14)	90	90	90
β, deg	93.642(13)	110.166(3)	105.08(2)	90
γ, deg	91.703(13)	90	90	120
V (Å^3)	881.8(3)	992.04(7)	846.1(4)	1845.17(12)
Z	2	4	4	6
d_{calc}, g/cm^3	1.293	1.394	1.415	1.384
μ, mm^{-1}	0.191	0.298	0.331	0.251
Total number of reflections	5113	4650	3054	10193
Number of independent reflections	3573	2398	1858	1590
R_{int}	0.0143	0.0247	0.0456	0.0401
Completeness, % (for Θ, deg)	99.2 (26.37)	97.4 (28.29)	95.8 (26.00)	99.3 (28.29)
Θ, deg	$2.89 < \Theta < 26.37$	$3.21 < \Theta < 28.29$	$2.65 < \Theta < 26.37$	$3.11 < \Theta < 28.29$
S (F^2)	1.001	1.000	1.007	1.000
Number of refined parameters	226	128	227	183
R_1 [$I > 2\sigma(I)$]	0.0365	0.0300	0.0565	0.0269
wR_2 [$I > 2\sigma(I)$]	0.0915	0.0599	0.0968	0.0530
R_1 (all reflections)	0.0647	0.0585	0.1276	0.0338
wR_2 (all reflections)	0.0972	0.0619	0.1086	0.0537
$\Delta e_{max/min}$, e Å^{-3}	0.213/–0.130	0.202/–0.209	0.613/–0.182	0.196/–0.146

packing of sulfoxide **VII** is interesting. Molecules **VII** in crystal are packed as two-chain bands oriented along the crystallographic *b* axis, and sulfoxide molecules in each chain are arranged contradirectionally. The chains are formed due to strong polar contacts $O^1\cdots S^1$ 2.936(2) Å [*x*, –1 + *y*, *z*], which are shorter by 0.384 Å than the sum of the corresponding van der Waals radii. In addition, a shortened molecular contacts $O^1\cdots S^8$ 2.906(2) Å [*x*, –1 + *y*, *z*] is observed (it is shorter by 0.314 Å than the sum of the corresponding van der Waals radii), indicating interaction between electrons on the oxygen atom and π-electron density of the C^8–S^1 bond. On the other hand, a fairly long distance between aromatic rings in the chain [4.383(2) Å for centroids and 4.210(2) Å for planes] excludes appreciable π–π interaction between molecules **VII**. The chains are linked together to form planar bands via "dimeric" contacts like $O^1\cdots S$ [1 – *x*, 1 – *y*, 1 – *z*] and $S^1\cdots O^1$ [1 – *x*, 1 – *y*, 1 – *z*] 3.193 Å that are shorter by 0.127 Å than the sum of the corresponding van der Waals radii. The principal parameters of X-ray diffraction experiment are collected in Table 1.

The ^{13}C NMR spectrum of compound **VIII** was consistent with the assumed structure. The ^{1}H NMR parameters of **VIII** were analogous to those reported in [3]. According to the X-ray diffraction data, compound

VIII crystallizes in $P2_1$ chiral space group (monoclinic crystal system) as two crystallographically independent molecules with fairly similar geometric parameters. The structure of one crystallographically independent molecule of **VIII** with atom numbering is shown in Fig. 3. Both molecules were assigned *R* configuration of the sulfur atom; however, small anomalous scattering effect did not allow us to unambiguously identify the absolute configuration of **VIII**. The bond lengths and bond angles in both molecules approach the corresponding standard values. Unlike compound **VII**, packing of molecules **VIII** in crystal is determined by intermolecular hydrogen bonds N–H···O–S which link them to non-centrosymmetric dimers. The hydrogen bond parameters are given in Table 2. As a result, dimer stacks oriented along the *b* axis are formed. Despite stack packing favorable for approach of aromatic benzimidazole fragments to each other, the corresponding interplanar distance equal to the length of the *b* edge [4.7081(13) Å] is too long for π–π interaction to occur.

The structure of compound **IX** was unambiguously determined by NMR spectroscopy and X-ray analysis. The ^{13}C NMR spectrum of **IX** differs from the spectrum of initial compound **IV** by downfield shift of the CH_2SO signal (δ_C 58.82 ppm), and the corresponding methylene protons resonate in the ^1H NMR spectrum of **IX** as two doublets at δ 4.36 and 4.56 ppm. The structure of molecule **IX** according to the X-ray diffraction data is shown in Fig. 4. Crystals of **IX** belong to $P6_2$ chiral space group in hexagonal crystal system. Molecule **IX** adopts a pincer-like conformation with closely located aromatic fragments [the torsion angle $C^1S^1C^8C^9$ is –62.12(5)°]. Helical packing of molecules **IX** in crystal (Fig. 5) is formed by chains twisted along the *c* axis. Molecules **IX** are linked through intermolecular hydrogen bonds between NH group and N^2 [$y + 1, -x + y + 1, z - 1/3$] in the benzimidazole fragments (Table 2) and strong polar contacts between sulfoxide groups $O^1\cdots S^1$ 3.173(2) Å [$y + 1, -x + y + 1, z - 1/3$], the latter being shorter by 0.147 Å than the sum of the corresponding van der Waals radii. On the whole, the geometric parameters of the above polar contacts are analogous to those found for compound **VII**.

Compound **X** was described previously [15]. Its ^{13}C NMR spectrum contained signals corresponding to the alkyl and aromatic fragments. The CH_2SO signal is observed at δ_C 56.58 ppm, and the corresponding proton signals appear as two multiplets at δ 2.84 and 2.91 ppm in the ^1H NMR spectrum.

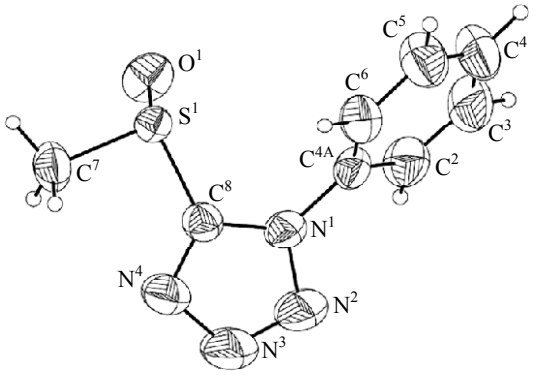


Fig. 2. Structure of the molecule of 5-methylsulfinyl-1-phenyl-1*H*-tetrazole (**VII**) according to the X-ray diffraction data. Non-hydrogen atoms are shown as thermal vibration ellipsoids with a probability of 50%.

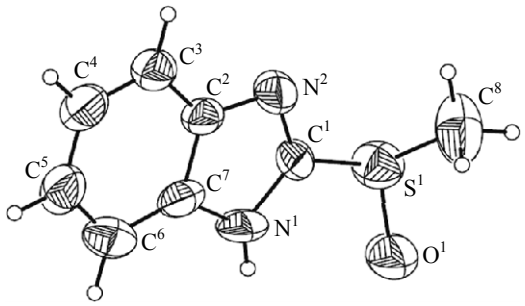


Fig. 3. Structure of the molecule of 5-methylsulfinyl-1*H*-benzimidazole (**VIII**) according to the X-ray diffraction data. Non-hydrogen atoms are shown as thermal vibration ellipsoids with a probability of 50%.

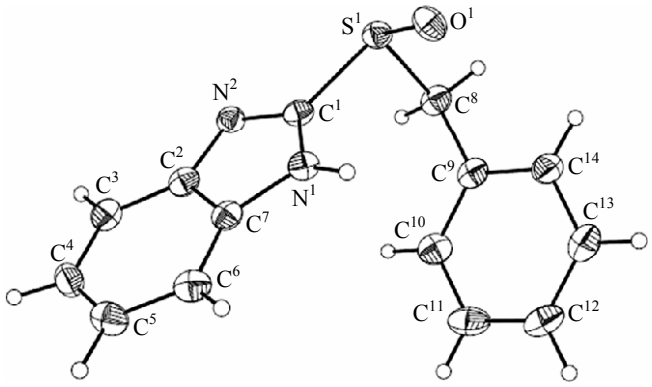


Fig. 4. Structure of the molecule of 5-benzylsulfinyl-1*H*-benzimidazole (**IX**) according to the X-ray diffraction data. Non-hydrogen atoms are shown as thermal vibration ellipsoids with a probability of 50%.

Thus the results of the present work showed that oxidation of polyfunctional sulfides with ClO_2 involves the sulfide sulfur atom with high chemoselectivity and gives the corresponding sulfoxides.

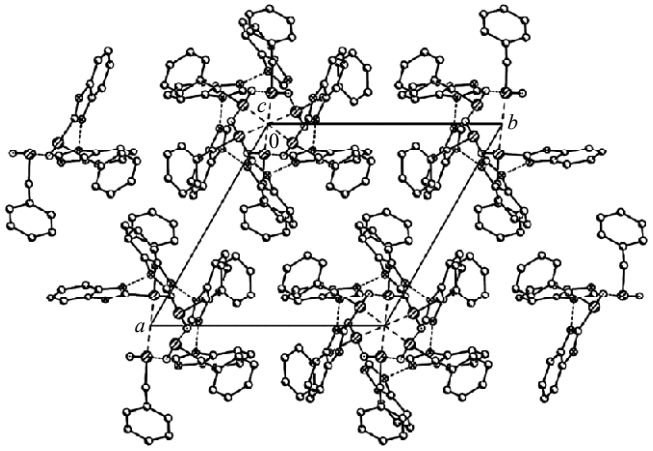


Fig. 5. A fragment of crystal packing of compound **IX**. Hydrogen atoms are not shown for the sake of simplicity.

EXPERIMENTAL

The IR spectra were recorded from solutions in carbon tetrachloride on Specord M80 and Prestige 21 spectrometers. The ^{1}H and ^{13}C NMR spectra were measured from solutions in $CDCl_3$ on Bruker DRX-400 (400 MHz) and Bruker Avance-II-300 (300 MHz) spectrometers. Gaz–liquid chromatography was performed on a Chrom-5 chromatograph equipped with a flame-ionization detector and a 2.5×3000-mm column packed with 6% of SKTF on Chromaton N-AW-HMDS; carrier gas helium. The oven temperature for analysis of sulfoxides was programmed from 50 to 250°C at a rate of 6 deg/min. The products were identified using authentic samples. Thin-layer chromatography was performed on Sorbfil plates using heptane–diethyl ether (1:2) as eluent; spots were developed by treatment with a 5% solution of potassium permanganate.

A solution of chlorine dioxide in an organic solvent was prepared by bubbling ClO_2 from an aqueous solution with a concentration *c* of 7 g/l through the corresponding solvent cooled to 0°C. Solvents were selected taking into account solubility of the initial sulfide: methylene chloride was taken for compounds **I**–**IV**, and ethanol, for sulfide **V**.

The X-ray diffraction data for compounds **I** and **VII**–**IX** were acquired according to standard procedure on an Xcalibur 3 automatic four-circle diffractometer equipped with a CCD detector (λMo*K*, graphite monochromator, ω-scanning, scan step 1.0°, frame time 15 s). No correction for absorption was introduced, taking into account its insignificance. The structures were solved by the direct method and were refined with respect to F^2 by the full-matrix least-squares procedure in anisotropic (isotropic for hydrogen atoms) approximation using SHELX97 software package [21]. Hydrogen atoms were visualized by difference electron density peaks and were taken into the refinement procedure according to the riding model with dependent thermal parameters. Table 1 contains the principal crystallographic parameters of compounds **I** and **VII**–**IX** and conditions of X-ray diffraction experiments. The complete sets of crystallographic data were deposited to the Cambridge Crystallographic Data Centre (entry nos. CCDC 767827–767830) and are available at *http://www.ccdc.cam.ac.uk/data_request/cif*.

3-Benzylsulfanyl-4,5-diphenyl-4*H*-1,2,4-triazole (I). Colorless crystals, mp 180–181°C; published data [22]: mp 150–151°C. ^{1}H NMR spectrum, δ, ppm: 4.51 s (2H, CH_2), 7.08–7.36 m (15H, C_6H_5). ^{13}C NMR spectrum, δ_C, ppm: 37.38 (CH_2), 126.71 (C^1_A), 127.36–129.84 (C_{arom}), 134.23 (C^1_B), 136.50 (C^1_C), 152.53 (C^3), 154.94 (C^5). Found, %: C 74.35; H 5.08; N 12.13; S 9.33. $C_{21}H_{17}N_3S$. Calculated, %: C 74.44; H 4.99; N 12.23; S 9.34.

Oxidation of sulfides I–V to sulfoxides VI–X (*general procedures*). *a*. An aqueous solution of chlorine dioxide, 3.6 ml (0.37 mmol, *c* = 7 g/l) was added over a period of 30 min under stirring at 20°C to a solution of 0.127 g (0.37 mmol) of compound **I** in 10 ml of methylene chloride. The mixture was extracted with methylene chloride, the extract was evap-

Table 2. Parameters of intermolecular hydrogen bonds in the crystalline structure of compounds **VIII** and **IX** according to the X-ray diffraction data[a]

Comp. no.	D–H	D–H, Å	H···A, Å	∠DHA, deg	D···A, Å	A
VIII	N^1–H^1	0.79(2)	2.06(2)	154(1)	2.797(2)	O^{1A} [–*x* + 1, *y* + 1/2, –*z* + 1]
	N^{1A}–H^{1A}	0.86(2)	1.99(2)	154(1)	2.795(2)	O^1 [–*x* + 1, *y* – 1/2, –*z* + 1]
IX	N^1–H^1	0.84(2)	2.03(2)	160(1)	2.839	N^2 [–*y* + 1, –*x* + *y* + 1, *z* – 1/3]

[a] The superscript "*A*" refers to the second crystallographically independent molecule of **VIII**.

orated, and the residue was subjected to column chromatography on silica gel using heptane–diethyl ether as eluent to isolate 0.118 g (89%) of 3-benzylsulfinyl-4,5-diphenyl-4*H*-1,2,4-triazole (**VI**). Colorless crystals, mp 197–198°C. IR spectrum: ν 1038 cm^{-1} (S=O). ^{1}H NMR spectrum, δ, ppm: 4.72 d and 5.05 d (1H each, CH_2, J = 12.46 Hz), 7.02–7.45 m (15H, C_6H_5). ^{13}C NMR spectrum, δ_C, ppm: 58.82 (CH_2), 125.64 (C_A^i), 127.55 (C_B^o), 128.61 (C_A^m), 128.68 (C_C^m), 128.76 (C_B^p), 128.97 (C_C^o), 129.91 (C_A^o), 130.39 (C_C^p), 130.49 (C_A^p), 130.88 (C_B^m), 132.90 (C_B^i, C_C^i), 138.31 (C^3), 155.99 (C^5). Found, %: C 70.12; H 4.85; N 11.60; O 4.35; S 9.00. $C_{21}H_{17}N_3OS$. Calculated, %: C 70.17; H 4.77; N 11.69; O 4.45; S 8.92.

b. A mixture of air with chlorine dioxide (evolved from an aqueous solution containing 0.5 mmol of ClO_2) was bubbled through a solution of 0.172 g (0.5 mmol) of compound **I** in 25 ml of methylene chloride over a period of 30 min at 20°C. The solvent was distilled off, and the residue was subjected to column chromatography on silica gel using heptane–diethyl ether as eluent to isolate 0.163 g (87%) of compound **VI**.

c. A solution of chlorine dioxide in methylene chloride, 9 ml (0.42 mmol), was added over a period of 30 min to a solution of 0.144 g (0.42 mmol) of compound **I** in 10 ml of methylene chloride under stirring at 20°C. The mixture was evaporated, and the residue was subjected to column chromatography on silica gel using heptane–diethyl ether as eluent to isolate 0.135 g (88%) of compound **VI**.

5-Methylsulfinyl-1-phenyl-1*H*-tetrazole (VII) [14]**.** Yield 92–97%. Colorless crystals, mp 79–80°C. IR spectrum: ν 1044 cm^{-1} (S=O). ^{1}H NMR spectrum, δ, ppm: 2.41 s (3H, CH_3), 7.55–7.72 m and 7.88–7.95 m (5H, C_6H_5). ^{13}C NMR spectrum, δ_C, ppm: 35.78 (CH_3), 130.25 (C^o), 132.36 (C^m, C^p), 133.87 (C^i), 142.46 (C^5).

2-Methylsulfinyl-1*H*-benzimidazole (VIII). Yield 85–89%. Colorless crystals, mp 139–140°C; published data [23]: mp 200–206°C. IR spectrum: ν 1055 cm^{-1} (S=O). ^{1}H NMR spectrum, δ, ppm: 3.23 s (3H, CH_3), 7.38 m (4H, C_6H_4), 12.33 s (1H, NH). ^{13}C NMR spectrum, δ_C, ppm: 41.54 (CH_3), 113.30 (C^3, C^6), 121.04 (C^4, C^5), 124.0 (C^2, C^7), 153.64 (C^1). Found, %: C 53.02; H 4.29; N 15.61; O 8.95; S 17.85. $C_{11}H_{12}N_2OS$. Calculated, %: C 53.31; H 4.47; N 15.54; O 8.88; S 17.79.

2-Benzylsulfinyl-1*H*-benzimidazole (IX) [24]**.** Yield 97–98%. Colorless crystals, mp 182.9–183.2°C. IR spectrum: ν 1050 cm^{-1} (S=O). ^{1}H NMR spectrum, δ, ppm: 4.36 d and 4.56 d (1H each, CH_2, J = 13.2, 11.6 Hz), 7.07 m (2H, 5-H, 6-H), 7.26 m (2H, 4-H, 7-H), 7.16 m (5H, C_6H_5), 7.57 s (1H, NH). ^{13}C NMR spectrum, δ_C, ppm: 58.82 (CH_2), 113.50 (C^4), 115.51 (C^7), 121.74 (C^5), 123.44 (C^6), 127.08 (C^m), 128.25 (C^o, C^p), 130.06 (C^i), 138.87 (C^{3a}, C^{7a}), 152.01 (C^2).

1-Butylsulfinyl-4-nitrobenzene (X). Yield 87–94%. Colorless liquid. IR spectrum: ν 1044 cm^{-1} (S=O). ^{1}H NMR spectrum, δ, ppm: 0.94 t (3H, CH_3, J = 7.2 Hz), 1.46 m (2H, 3′-H), 1.80 m (2H, 2′-H), 2.84 m and 2.91 m (1H each, 1′-H), 7.83 d and 8.40 d (2H each, C_6H_4, J = 8.4, 8.8 Hz). ^{13}C NMR spectrum, δ_C, ppm: 13.42 ($C^{4'}$), 21.62 ($C^{3'}$), 23.65 ($C^{2'}$), 56.58 ($C^{1'}$), 124.10 (C^3, C^5), 124.97 (C^2, C^6), 149.23 (C^4), 151.53 (C^1).

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