Article

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Structural Features of Certain *p*-substituted Phenyl 2-nitrovinyl Sulfones

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The structure of *p*-substituted phenyl 2-nitrovinyl sulfones was studied through different spectral methods. It has been found that the nucleophilic substitution reactions of sodium phenylsulfinates with 1-chloro-2-nitroethene occur stereospecifically. The formation of *E*-isomers of this type of substituted phenyl vinyl sulfones was confirmed by FTIR and NMR spectroscopy. It was suggested that the *E*-isomers formation was determined by the more stable conformation of the carbanionic intermediate as a result of intramolecular rotation around single Cα-Cβ bond. This appears to be the product-determining pathway during the interaction of sodium phenyl sulfinates with the highly activated substrate 1-chloro-2-nitroethene.

Keywords: Sulfinate *S*-vinylation; Phenyl 2-nitrovinyl sulfones; Nucleophilic vinylic substitution; Substituent effects; *E*-isomers.

INTRODUCTION

The interaction of sulfinic acids or their salts with a wide variety of carbon-centered electrophiles such as vinyl and aryl halides results predominantly in the formation of a new carbon-sulfur bond, and constitutes the most important method for the synthesis of sulfones.[1-3] Irrespective of the fact that the organic sulfinic acids and sulfinate anions exhibit a typical ambident reactivity,[4] in polar protic solvents such as aqueous or aqueous-alcoholic medium, the oxygen atom is blocked due to the formation of hydrogen bonds.[2,3] Thus, the sulfur atom is the only nucleophilic center, and even in the reactions with the less electrophilic carbenum ions, the sulfur-centered attack is prevalent.[4]

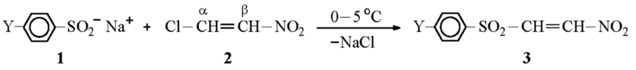
A number of researchers have studied the interaction of phenylsulfinic acids with alkenes, bearing electron-withdrawing groups such as nitro- and cyano-groups, i.e. with alkenes behaving as Michael acceptors. The presence of good leaving groups as halogen atoms in β-position relative to the conjugated nitro group determines their high activity towards nucleophilic reagents.[5,6–11] In addition, the interaction of arenesulfinates with *α,β*-unsaturated compounds containing vinyl-bound halogen can be accomplished by different mechanisms, depending on pH of the reaction medium.[2,3,10] Thus, the aryl haloalkyl sulfones were found to be formed in slightly-acidic medium. However, in slightly-alkaline medium, the primary addition reaction is followed by an elimination process, resulting in the formation of β-substituted aryl vinyl sulfones.[3,10,11]

The aim of the present study was to examine the structural characteristics of series of *p*-substituted phenyl 2-nitrovinyl sulfones prepared by known method under mild conditions. Accordingly, a complete analysis by employing different spectral methods was undertaken, in order to establish the correct configuration of the resulting phenyl vinyl sulfones.

RESULTS AND DISCUSSION

The interaction of *p*-substituted sodium phenylsulfinates (1) with 1-chloro-2-nitroethene (2) was conducted in aqueous medium, and at relatively low temperature as shown in Scheme 1.

Scheme 1 Synthesis of *p*-substituted phenyl 2-nitrovinyl sulfones



Y	H	*p*-CH_3	*p*-Cl	*p*-I	*p*-OCH_3	*p*-NO_2
3	**a**	**b**	**c**	**d**	**e**	**f**

The implementation of the reaction in a polar solvent and slightly-alkaline medium (pH 7.2–7.4) suggests the formation of aryl 2-nitrovinyl sulfones (Scheme 1, compounds 3a–3f). The predominant formation of carbon–sulfur bond can be explained by the greater softness of sulfur atom as a nucleophile, compared to that of oxygen, the sul-

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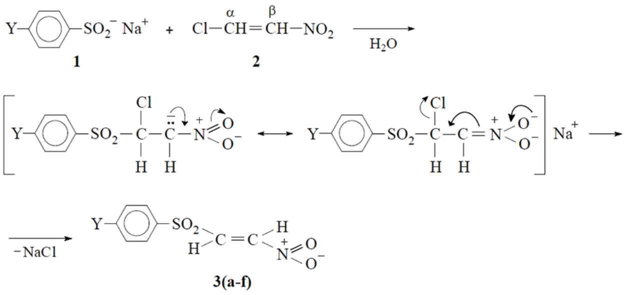
fur atom being preferably bound to the very soft electrophilic center ($C\alpha$-atom) in 1-chloro-2-nitroethene. According to the reactivity indices of 1-chloro-2-nitroethene calculated by MOPAC 93 program,[12] the electrophilicity of $C\alpha$-atom is higher compared to that of $C\beta$-atom. This is due to the higher positive values of the acceptor superdelocalizability ($S\mu^N$) of the $C\alpha$-atom as well as to the fact that the negative charge (Q) of the $C\alpha$-atom is about three times as low as that of the $C\beta$-atom, regardless of the soft electrophilicity of both the $C\alpha$ and $C\beta$. For example, $S\mu^N(C\alpha)$ parameter amounts to 0.2710 $(a.u.)^2/eV$, compared to $S\mu^N(C\beta)$ which is 0.2425 $(a.u.)^2/eV$. At the same time, the net atomic charge of $Q(C\alpha)$ is -0.068 a.u. *vs.* that of $Q(C\beta)$ equal to -0.212 a.u.

In fact, 1-chloro-2-nitroethene represents *α,β*-unsymmetrically disubstituted substrate, in which the double bond is affected by the inconsistent action of two electron-withdrawing groups,[13,14] with the predominant influence of both the negative inductive and resonance effects of the nitro group. This substrate should be referred to, as a highly-activated system, according to the classification of *Rappoport*.[15] The calculated p*K*a value of the *β*-H atom in the chloronitroethene fragment ($=CH-NO_2$) is 19.03, which is consistent with the requirement for p*K*a = 10–20 for highly-activated systems.

According to many authors,[13-17] the substitution of halogen atoms in activated haloalkenes proceeds as a two-step process with a formation of carbanionic intermediate. The so-called nucleophilic vinylic substitution (S_NV) that includes reactions of addition-elimination (Ad_N-E) predominates for compounds, containing electron-withdrawing substituents[15] such as NO_2 group. The S_NV mechanism for the interaction of *p*-substituted phenylsulfinates with 1-chloro-2-nitroethene is presented in Scheme 2.

Scheme 2 S_NV (Ad_N-E) mechanism for the interaction of *p*-substituted phenylsulfinates with 1-chloro-2-nitroethene



Y = H (3a); CH_3 (3b); Cl (3c); I (3d); OCH_3 (3e); NO_2 (3f)

The presence of a strong *π*-electron acceptor such as the nitro group at *β*-position towards the electrophilic center (i.e., the $C\alpha$-atom in 1-chloro-2-nitroethene) results in an increase of the rate of carbanion formation as well as its stability. This is due to the potential of delocalization of the negative charge[14,16] involving the nitro group, as shown in Scheme 2.

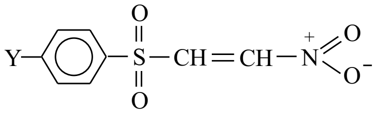
Both the composition and the structures of the *p*-substituted phenyl-2-nitrovinyl sulfones obtained have been determined by using microanalytical and spectral methods (Tables 1-5). As shown in Table 1, sulfones were synthesized with moderate to high yields (76-94%).

Apparently, the difference in the yields of sulfones 3a-3f is determined by the electronic effects of the substituents and their influence on the nucleophilicity of sulfur atom. This phenomenon is most pronounced with the *p*-methoxy group as compound 3e, which enhances the nucleophilicity of sulfur significantly, due to the predominant positive resonance effect of the substituent (Table 1, yield 94%). On the other hand, the strong electron-withdrawing effect of the *para*-nitro group likely has an influence on the internal resonance effect of the sulfonyl group. This probably increases the nucleophilicity of the sulfur atom and facilitates the formation of $S-C_\alpha$ bond (Table 1, compound 3f). The yields were little affected by the introduction of methyl group which has a slightly pronounced effect of hyperconjugation and chlorine atom which apparently do not have noticeable effect on the the nucleophilicity of sulfur atom. However, due to the low electronegativity of iodine atom, compound 3d was prepared in a high yield (91%).

The characteristic frequencies of the absorption bands in the FTIR spectra of the compounds studied in KBr pellets are shown in Table 2.

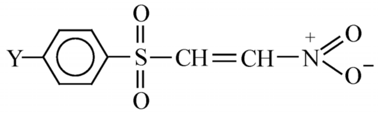
The type of substitution in the aromatic ring was confirmed by the out-of-plane deformation vibrations of the aromatic hydrogen atoms (γ(Ph-H)) registered within 820–805 cm^{-1}. The conjugated nitro group in the compounds studied was confirmed by the strong absorption bands within the 1528–1525 and 1343–1337 cm^{-1} regions, characteristic for $\nu as(NO_2)$ and $\nu s(NO_2)$, respectively. The presence of sulfonyl group in sulfones in solid state is characterized by the strong absorption bands at 1325–1310 ($\nu as(SO_2)$) and 1152–1140 cm^{-1} ($\nu s(SO_2)$).[18] The frequency of the stretching vibrations of this group is almost not affected by the nature of substituents at *p*-position in the benzene ring. The sulfonyl group bound to the aromatic

Table 1. Physical and analytical data of *p*-substituted phenyl 2-nitrovinyl sulfones



No	Y	Yield (%)	Tm (°C)	Formula Mol. wt.	Analysis (%) found (calcd)			
					C	H	N	S
3a	H	72	175	$C_8H_7NO_4S$ 213.21	45.07 (45.27)	3.31 (3.38)	6.57 (6.31)	15.04 (15.04)
3b	CH_3	76	120	$C_9H_9NO_4S$ 227.24	47.57 (47.29)	3.99 (4.79)	6.16 (5.82)	14.11 (14.79)
3c	Cl	78	130	$C_8H_6ClNO_4S$ 247.66	40.93 (38.88)	2.44 (2.25)	5.66 (5.13)	12.95 (13.34)
3d	I	91	180	$C_8H_6INO_4S$ 339.11	28.33 (27.95)	1.78 (1.59)	4.13 (3.95)	9.46 (9.98)
3e	OCH_3	94	130	$C_9H_9NO_5S$ 243.24	44.44 (41.12)	3.73 (3.70)	5.76 (5.65)	13.18 (13.46)
3f	NO_2	89	170	$C_8H_6N_2O_6S$ 258.21	37.21 (37.54)	2.34 (2.02)	10.85 (9.83)	12.42 (12.56)

Table 2. Wave numbers ($\tilde{\nu}$, cm^{-1}) and vibration modes of characteristic absorption bands in the FTIR spectra of *p*-substituted phenyl 2-nitrovinyl sulfones



No	Y	ν(Ph-H)	ν(C=C) (arene ring)	$\nu(NO_2)$		$\nu(SO_2)$		$\nu(Ph\text{-}SO_2)$	ν(C=C) *E*-isomer	γ(=C-H) *E*-isomer	δ(Ph-H)	γ(Ph-H)
				as	s	as	s					
3a	H	3092 3070 3027	1575 1460 1445	1525	1337	1310	1143	1077	1623	935	1183 1017	753 728
3b	CH_3	3093 3073	1590 1504 1492	1528	1347	1317	1145	1081	1624	939	1190 1017	811
3c	Cl	3100 3057	1577 1504 1471	1528	1343	1325	1152	1083	1626	939	1187 1007	820
3d	I	3092 3060	1560 1471 1453	1525	1340	1315	1141	1079	1624	935	1183 1004	812
3e	OCH_3	3093 3057	1590 1504 1490	1527	1343	1313	1140	1080	1624	935	1187 1015	805
3f	NO_2	3100 3060	1572 1504 1470	1527	1341	1324	1151	1081	1625	939	1183 1009	817

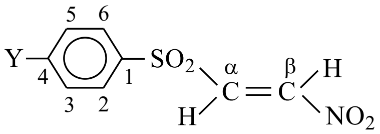
ring was also identified by the absorption band at 1083–1077 cm^{-1}. According to some authors, this band is associated with the stretching vibrations of the C(aryl)-S bond.[19,20]

The presence of C=C bond in the nitrovinyl fragment of the sulfones studied was detected by the absorption band at 1625 cm^{-1}. The shift of this band towards the lower frequencies proves the well-pronounced π,π-conjugation, involving both the C=C bond and $-NO_2$ group. The double bond was also confirmed by the strong absorption at 939-935 cm^{-1} (Table 2), which is characteristic for the out-of-plane deformation vibrations of the =Csp^2-H bond in disubstituted alkenes of E-configuration.[21,22]

The structure of sulfones obtained was also confirmed by ^{1}H and ^{13}C NMR spectroscopy studies. The chemical shift values for the hydrogen and carbon atoms in the benzene ring and the vinyl fragment are presented in Tables 3 and 4, respectively.

The aromatic protons in the *p*-disubstituted benzene ring were registered within the δ = 7.44-7.97 *ppm* interval (Table 3). For compounds 3b, 3c and 3d, the *ortho*-protons towards the SO_2 group produce resonance signals in a weaker field, compared to the *meta*-protons. This is apparently due to the electron-withdrawing effects of the sulfonyl group. When the *p*-substituents are NO_2 and OCH_3 groups, the deshielding of *meta*-protons is more pronounced, which is probably determined by the asynchronous influence of the negative inductive effects of the NO_2, OCH_3 и SO_2 groups. In addition, the proton interaction constant values of the hydrogen atoms in positions 2 and 3, and 5 and 6, respectively, i.e. the parameter *J*ortho varied within 8.0-8.9. These values were found to be higher as substituents with predominantly negative inductive effect such as chlorine atom (3c) and nitro group (3f) were located in the *p*-position of the benzene ring. On the other hand, the vicinal coupling constant values *J*(H*α*-H*β*) in the nitrovinyl fragment were changed negligibly (e.g., 13.0-

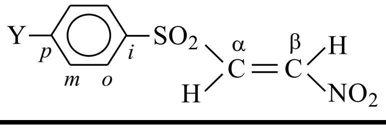
Table 3. ^{1}H NMR Chemical shifts of *p*-substituted phenyl 2-nitrovinyl sulfones



No	Y	δ (benzene ring), ppm		*Jortho* Hz	δ (vinyl fragment), ppm		*J*(H*α*-H*β*) Hz
		ortho-H	*meta*-H		H*α*	H*β*	
3a	H[a]	7.95–8.00 (m, 2H)	7.62–7.70 (m, 2H)	-	7.59 (d, 1H)	7.68 (d, 1H)	13.1
3b	CH_3[b]	7.84 (d, 2H)	7.44 (d, 2H)	8.0	7.58 (d, 1H)	7.65 (d, 1H)	13.0
3c	Cl	7.91 (d, 2H)	7.64 (d, 2H)	8.9	7.57 (d, 1H)	7.69 (d, 1H)	13.0
3d	I	7.97 (d, 2H)	7.62 (d, 2H)	8.1	7.54 (d, 1H)	7.64 (d, 1H)	13.0
3e	OCH_3[b]	7.44 (d, 2H)	7.84 (d, 2H)	8.3	7.57 (d, 1H)	7.65 (d, 1H)	13.0
3f	NO_2	7.63 (d, 2H)	7.91 (d, 2H)	8.8	7.57 (d, 1H)	7.69 (d, 1H)	13.0

[a] *δ* = 7.74–7.81 ppm (m, 1H, *para*-H); [b] *δ* = 2.49 ppm (s, 3H, CH_3).

Table 4. ^{13}C NMR Chemical shifts of *p*-substituted phenyl 2-nitrovinyl sulfones



No	Y	*δ*, ppm					
		ortho-C	*meta*-C	*para*-C	*ipso*-C	Cα-SO_2	Cβ-NO_2
3a	H	128.6	130.0	135.3	137.5	138.0	146.3
3b	CH_3[a]	128.6	132.1	146.9	134.5	138.3	145.9
3c	Cl	130	130.4	142.5	135.9	137.6	146.6
3d	I	129.7	139.4	104.1	137.2	137.6	146.6
3e	OCH_3[a]	128.5	130.6	134.4	146.9	138.3	145.9
3f	NO_2	130.0	130.4	135.9	146.6	137.7	142.5

[a] *δ* = 21.8 ppm (CH_3).

13.1) for all sulfones synthesized. This is in accordance with the prerequisite for *E*-configuration of disubstituted alkenes, according to which, the $J(H\alpha\text{-}H\beta)$ value should vary within 11(12)-18 Hz in such a case.[21,22]

As seen from Table 3, the difference in the chemical shift of the α- and β-protons of the vinyl fragment is, approximately, 0.1 *ppm*. Generally, the resonance signals of the β-protons ($H\beta$) are registered in a slightly weaker field than those of α-protons ($H\alpha$). This is apparently due to the more pronounced electron-withdrawing effect of the nitro group, compared to the sulfonyl one. This difference is particularly well-pronounced with the chemical shifts of the carbon atoms ($C\alpha$ and $C\beta$) in the ^{13}C NMR spectra (Table 4), which are directly associated with the SO_2 и NO_2 groups, respectively. Only for the compound 3f, containing nitro group in *para*-position in the benzene ring, the deshielding of the $C\beta$-atom was less pronounced, compared to the rest of sulfones. This is probably associated with the incoherent electron-withdrawing effects of both nitro groups, located in the β- and *p*-positions in the vinyl fragment and benzene ring, respectively.

Strong absorption bands were also registered in the UV–spectra of the compounds studied in two regions with maximums at 218-237 *nm* and 248-291 *nm*, respectively (Table 5, Figure 1) with molar absorbance values ($a\nu$) higher than 10000 dm^3 mol^{-1} cm^{-1} ($\lg a\nu \geq 4$). The absorption maximums were without pronounced vibration features, which indicated the conjugated structure of these chemicals.[21,22]

The specific influence of substituents (compounds 3b-3f) on the positions of the absorbance maximums is determined by the degree of their conjugation with the π-electrons of the aromatic ring. The following features could be noted, depending on the nature of the substituent(s): (*i*) methyl group elicits negligible bathochromic effect in the first and the second absorbance regions (Table 5, compound 3b) which is obviously due to the slightly pronounced effect of hyperconjugation; (*ii*) significant difference in the influence of chlorine and iodine atoms on the batochromic shift of λ_{max} was observed within the second

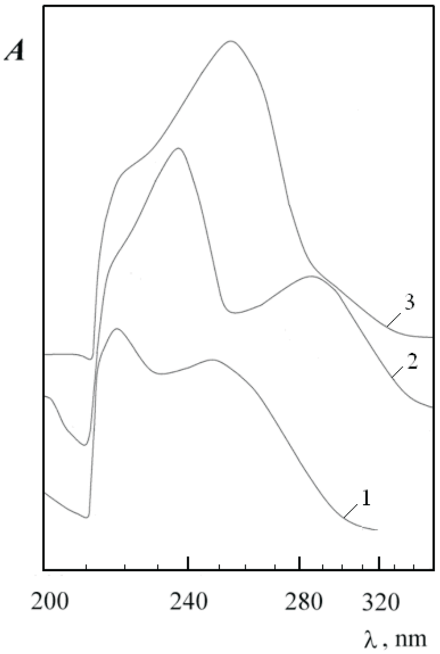
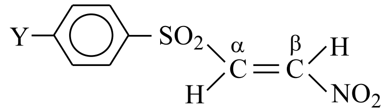


Fig. 1. UV spectra of phenyl 2-nitrovinyl sulfone (1), *p*-methoxyphenyl 2-nitrovinyl sulfone (2) and *p*-nitrophenyl 2-nitrovinyl sulfone (3).

Table 5. Absorption maximum in the near UV-absorption region of *p*-substituted (*E*)-phenyl 2-nitrovinyl sulfones (solution in dioxan)



No	Y	First absorption region			Second absorption region		
		λ_{max} (nm)	$\Delta\lambda$[a] (nm)	lg *a*ν	λ_{max} (nm)	$\Delta\lambda$[a] (nm)	lg *a*ν
3a	H	218	-	4.13	248	-	4.10
3b	CH_3	223	5	4.40	258	10	4.24
3c	Cl	223	5	4.25	258	10	4.04
3d	I	234	16	4.16	283	35	4.07
3e	OCH_3	237	19	4.18	291	43	3.92
3f	NO_2	-	-	-	256	8	4.31

[a] $\Delta\lambda$ - the difference between λ_{max} of *p*-substituted (*E*)-phenyl 2-nitrovinyl sulfones and λ_{max} of non-substituted phenyl 2-nitrovinyl sulfone.

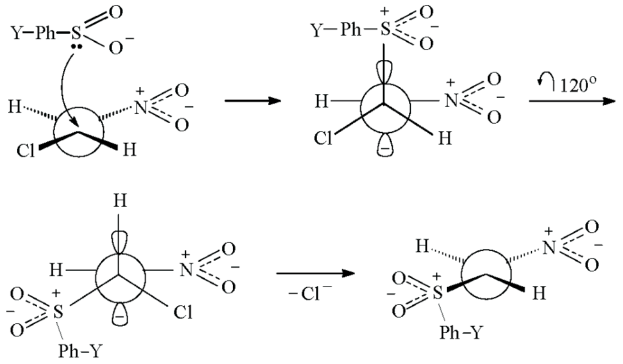
absorbance region, associated with strongly pronounced negative inductive effect of chlorine as compared to that of iodine;[22,23] (*iii*) the batochromic shift of λ_{max} was mostly pronounced with the methoxy group, especially, in the second absorbance region (Table 5, $\Delta\lambda = 43$ nm). It could be assumed that intramolecular charge transfer occurred as a result from the coherent conjugation in the *para*-disubstituted aromatic fragment and the nitrovinyl moiety in the molecule; (*iv*) the formation of an extended maximum within the 220–270 *nm* region for 4-nitrophenyl 2-nitrovinyl sulfone (Figure 1, curve 3) is probably determined by the presence of two NO_2-groups in both the benzene ring and nitrovinyl moiety, respectively. In this case, as a result from the incoherent conjugation along the molecule, the batochromic shift is insignificant ($\Delta\lambda = 8$ *nm*). However, since the molar absorbance is rather high (av = 20426 dm^3 mol^{-1} cm^{-1}), it could be inferred that a local charge transfer, involving the nitrophenyl and nitrovinyl molecular fragments takes place, as depicted by the resonance structures 1 and 2 shown in Scheme 3.

The strong maximum at 256 *nm* of 4-nitrophenyl 2-nitrovinyl sulfone (Fig. 1, curve 3) is probably associated not only with π,π–conjugation, involving the corresponding molecular fragments but, also, with the electronic transitions localized within the nitro groups.

Based on the spectral studies such as FTIR and ^{1}H NMR, it was confirmed that the synthesized sulfones possess *E*-configuration. The formation of *E*-isomers by the interaction of *p*-substituted phenylsulfinates with 1-chloro-2-nitroethene is shown in Scheme 4. Particularly stable conformation of the carbanionic intermediate is obtained by rotation at 120° counter-clockwise. This corresponds to the studies of a number of authors on the S_NV-reactions with various substrates and other nucleophilic reagents.[14-17,23,24]

It could be assumed that the principal reason for the preferred formation of *E*-isomers of phenyl 2-nitrovinyl sulfones is associated with the conformational stability of the α-phenylsulfonyl β-nitrocarbanionic intermediate. The stereochemical result corresponds to the formation of sterically less hindered *E*-isomers, particularly, with a bulky nucleophilic reagent such as the arenesulfinate anion. Their formation is additionally facilitated by the possibility of intramolecular rotation around single $C\alpha$–$C\beta$ bond, owing to the reduced rotational barrier due to a conjugation between the carbanionic 2*p*-orbital and the π-orbital of nitro group.[16,24] Moreover, as a result of the internal rotation, the electrostatic repulsion between the dipoles of sulfonyl group and nitro group in the stabilized carbanionic intermediate is precluded (Scheme 4).

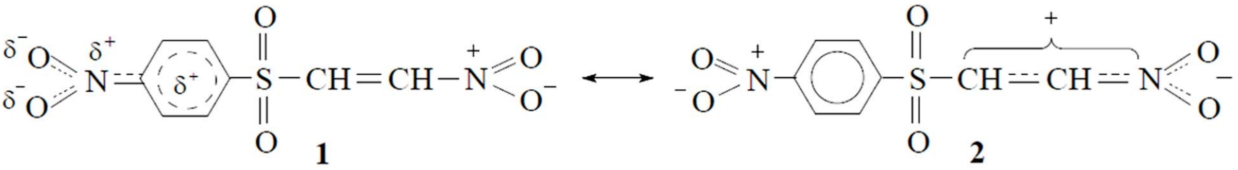
Scheme 4 Formation of E-isomers by interaction of *p*-substituted phenylsulfinates with 1-chloro-2-nitroethene, according to a two-step carbanionic S_NV-mechanism.



EXPERIMENTAL

General: The compounds synthesized were characterized by elemental analysis, FTIR spectroscopy, ^{1}H– and ^{13}C–NMR spectroscopy, as well as UV spectroscopy. The microanalyses were performed using a *Carlo Erba 1104* instrument (Italy). Fourier transform IR spectra were run on *Tensor 27* FTIR spectrometer (Germany) as KBr pellets. The spectra were scanned in the range 4000–400 cm^{-1}. ^{1}H and ^{13}C NMR spectra of phenyl 2-nitrovinyl sulfones were recorded on a *Bruker Avance II+* 600 (600 MHz) equipped with double ^{1}H/^{13}C measuring head (δ in ppm and *J* in Hz). The product samples were dissolved in $CDCl_3$ and TMS was used as an internal standard. The UV absorption of

Scheme 3 Possible resonance structures due to the π,π-conjugation in the structural fragments of 4-nitrophenyl 2-nitrovinyl sulfone



phenyl 2-nitrovinyl sulfones studied was determined at room temperature in an ethanol medium by means of spectrophotometer *Boeco Model S-20* (Germany). The melting temperatures (Tm) were determined using MOM Derivatograph OD-102 (Hungary) at a heating rate of 6 °C/min.

General procedure for preparation of *p*-substituted phenyl 2-nitrovinyl sulfones

Preparation of p-substituted phenylsulfinic acids and 1–chloro–2–nitroethene: All the substituted and non-substituted sodium phenylsulfinates were prepared by known procedure, employing reduction of the corresponding phenylsulfonyl chlorides with excess of sodium sulfite.[24,25] The corresponding sodium salts of arenesulfinic acids were obtained by adding an equimolar amount of 30% NaOH to water-alcoholic solution of the acids at 0–5 °C until neutral reaction was reached. Following the evaporation of solvents, the crystalline mass was filtered and rinsed with absolute ethanol and ether.

1–Chloro–2–nitroethene was prepared by addition reaction of NO_2Cl (or liquefied NO_2 and Cl_2) to vinyl chloride at –30 ÷ -40 °C in CCl_4 medium, followed by dehydrochlorination of the 1,1-dichloro-2-nitroethane thus obtained by CH_3COONa in dry ether medium. The compounds were characterized by determining their physical constants (1,1-dichloro-2-nitroethane: b.p. 60–70 °X/10 mm, n_D^{20} 1.4695; 1-chloro-2-nitroethene: b.p. 42–43 °X/30 mm; n_D^{20} 1.4900).

Interaction of sodium phenylsulfinates with 1–chloro–2–nitroethene: The interaction of sodium phenylsulfinates with 1-chloro-2-nitroethene was carried out according to the previously described procedure.[11] To an emulsion of 0.01 mol 1-chloro-2-nitroethene in water, 0.01 mol of freshly prepared solution of 0.01 mol sodium salt of the corresponding phenylsulfinic acid in 100 cm^3 water were added at 0–5 °C during 30 min under stirring. The pH of the reaction medium was maintained at 7–7.5. The crystalline mass was then filtered and rinsed repeatedly with distilled water. After drying, the products were purified by recrystalization using toluene.

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