

# Nitrochlorination of Methyl Tricyclo[4.1.0.0<sup>2,7</sup>]heptane-1-carboxylate and Phenyl Tricyclo[4.1.0.0<sup>2,7</sup>]hept-1-yl Sulfone

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**Abstract**—Treatment of methyl tricyclo[4.1.0.0<sup>2,7</sup>]heptane-1-carboxylate and phenyl tricyclo[4.1.0.0<sup>2,7</sup>]hept-1-yl sulfone with a ~1:8 mixture of N<sub>2</sub>O<sub>4</sub> and NOCl in diethyl ether at –5 to 0°C gave products of formal *anti*-addition of NO<sub>2</sub>Cl at the central C<sup>1</sup>–C<sup>7</sup> bond. In the reaction with phenyl tricyclo[4.1.0.0<sup>2,7</sup>]hept-1-yl sulfone nitryl chloride acts as an effective chlorinating agent; as a result, a mixture of diastereoisomeric *syn*- and *anti*-6,7-dichlorobicyclo[3.1.1]hept-6-yl phenyl sulfones at a ratio of 7.5:1 is formed.

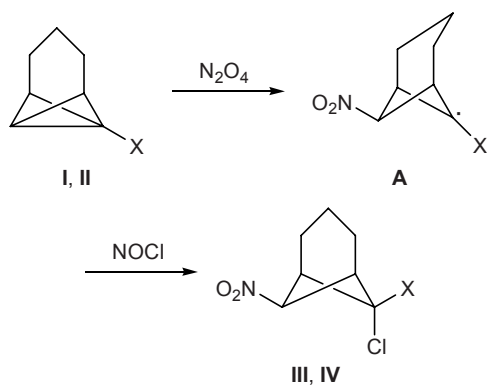
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In the preceding communication [1] we showed that tricycloheptanes **I** and **II** react with dinitrogen tetroxide, which behaves as ambident NO<sub>2</sub> radical, to give addition products at the central C<sup>1</sup>–C<sup>7</sup> bond, the corresponding 6,7-dinitro and 6-nitro-7-nitrosooxy derivatives. While performing these studies we found that the composition of products obtained from compounds **I** and **II** became more complex when dinitrogen tetroxide was purified using CaCl<sub>2</sub> instead of P<sub>2</sub>O<sub>5</sub> [2]; in each case, an additional chlorine-containing component was present in the reaction mixture. We presumed that the source of chlorine is nitrosyl chloride generated from N<sub>2</sub>O<sub>4</sub> and CaCl<sub>2</sub> via exchange reaction. In order to verify this assumption, we synthesized nitrosyl chloride by reaction of gaseous N<sub>2</sub>O<sub>4</sub> with anhydrous

KCl according to the procedure described in [3], and its mixture with N<sub>2</sub>O<sub>4</sub> at a ratio of ~8:1 was brought into reaction with compounds **I** and **II** in diethyl ether at –5 to 0°C. The only product formed in the reaction with ester **I** was nitro chloride **III** (Scheme 1). Sulfone **II** reacted with NOCl/N<sub>2</sub>O<sub>4</sub> to give a mixture of two compounds at a ratio of 10:1. The major product, nitro chloride **IV**, was isolated as individual substance, while the minor product was not identified. Just chlorides **III** and **IV** were the above additional products formed in the reactions of **I** and **II** with N<sub>2</sub>O<sub>4</sub> preliminarily purified using CaCl<sub>2</sub>.

The structure of compounds **III** and **IV** is fairly consistent with their <sup>1</sup>H and <sup>13</sup>C NMR spectra. Considerably lower chemical shifts of the C<sup>6</sup> atom in the <sup>13</sup>C NMR spectra of **III** and **IV** as compared to model dinitro ester and dinitro sulfone [1] indicate the presence of a chlorine atom rather than nitro group (which is characterized by the strongest α-effect [4]) in the geminal position with respect to the X substituent. The configuration at the C<sup>7</sup> atom follows from the <sup>1</sup>H NMR spectra which contain a triplet signal from the *anti*-7-H proton [5]. The *exo* orientation of the chlorine atom at C<sup>6</sup> in **III** and **IV** is confirmed by similar chemical shifts of the 7-H protons in the <sup>1</sup>H NMR spectra of these compounds. An additional support for the assumed configuration at C<sup>6</sup> is provided by downfield shift of the 1-H/5-H and 2-H/4-H signals in the spectrum of sulfone **IV** relative to the corresponding sig-

**Scheme 1.**

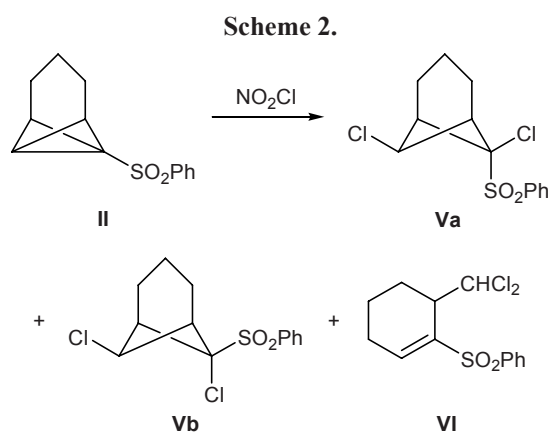


**I, III, X = MeOCO; II, IV, X = PhSO<sub>2</sub>.**

nals of ester **III**. This is explained by stronger long-range deshielding effect of the sulfonyl group as compared to alkoxy carbonyl substituent [6].

Presumably, like the nitration process [1], the nitrochlorination follows radical addition mechanism. The reaction is initiated by NO<sub>2</sub> radical generated from N<sub>2</sub>O<sub>4</sub>, and nitrosyl chloride acts as chlorine carrier.

It should be noted that intermediate 6-norpinanyl radical **A** takes up chlorine atom preferentially at the spatially more accessible *exo* side, thus leading to *anti*-adducts **III** and **IV**. An alternative path for formation of compounds **III** and **IV** could involve preliminary oxidation of nitrosyl chloride with dinitrogen tetroxide to give nitryl chloride NO<sub>2</sub>Cl, which could add to tricycloheptane **I** or **II** according to radical mechanism [7]. We examined reactions of ester **I** and sulfone **II** with nitryl chloride prepared by treatment of chlorosulfonic acid with a mixture of oleum and fuming nitric acid. From ester **I** we obtained a complex mixture of polychlorinated products which we failed to isolate and identify. Sulfone **II** reacted with NO<sub>2</sub>Cl in chloroform at -5 to 0°C to give a mixture of three dichloro derivatives **Va**, **Vb**, and **VI** at a ratio of 17:2.5:1 (Scheme 2). When the reaction was carried out in diethyl ether, the products were only stereoisomeric dichlorides **Va** and **Vb** at a ratio of 7.5:1 (according to the GLC and <sup>1</sup>H NMR data). In no case nitro chloride **IV** was detected. Compounds **Va** and **Vb** were also synthesized by independent method, i.e., by reaction of sulfone **II** with (dichloro-λ<sup>3</sup>-iodanyl)benzene in carbon tetrachloride at 20°C under UV irradiation.\*



Compounds **Va** and **VI** were isolated as individual substances by column chromatography on Al<sub>2</sub>O<sub>3</sub>, fol-

\* We found that treatment of sulfone **II** with molecular chlorine in CCl<sub>4</sub> at 0°C gives mainly dichloride **VI** and seven minor products (GLC data) which were not identified (see Experimental).

lowed by recrystallization. Sulfone **Vb** was characterized as a mixture with diastereoisomer **Va** (stereochemical purity 65%). The norpinane structure and configuration of the C<sup>7</sup> atom in compounds **Va** and **Vb** were unambiguously determined by <sup>1</sup>H and <sup>13</sup>C NMR spectroscopy. The configuration of C<sup>6</sup> was assigned by comparing the spectral parameters of **Va** and **Vb** with those of model bromine-containing analogs which were reported previously [8]. The structure of **Va** was unambiguously proved by X-ray analysis [9]. Cyclohexenyl sulfone **VI** was identified on the basis of the IR and NMR spectra. The IR spectrum of **VI** contained an absorption band at 1643 cm<sup>-1</sup> due to stretching vibrations of the double C=C bond. The olefinic proton in **VI** resonated in the <sup>1</sup>H NMR spectrum as a triplet at δ 7.4 ppm, the CHCl<sub>2</sub> proton gave a doublet at δ 6.6 ppm, and the corresponding carbon signal was located at δ<sub>C</sub> 74.8 ppm in the <sup>13</sup>C NMR spectrum.

Presumably, dichlorides **Va** and **Vb** are formed in the reaction of sulfone **II** with nitryl chloride according to radical addition mechanism where NO<sub>2</sub>Cl acts as halogenating (chlorinating) agent in a way similar to the action of CCl<sub>4</sub> (chlorination), CBrCl<sub>3</sub> or CBr<sub>4</sub> (bromination), CCl<sub>3</sub>I (iodination) [10, 11], and benzenesulfonyl bromide (bromination) [8] toward bicyclobutane compounds. The formation of monocyclic dichloride **VI** together with dichloronorpinanes **Va** and **Vb** in the reaction of sulfone **II** with NO<sub>2</sub>Cl in CHCl<sub>3</sub> may be rationalized assuming isomerization of intermediate *endo*-7-chloro-6-phenylsulfonylnorpinan-6-yl radical into chloro(2-phenylsulfonylcyclohex-2-en-1-yl)methyl radical, which is analogous to cyclobutyl-homoallyl rearrangement [11, 12]. A probable reason for the formation of a more complex mixture of products in the reaction of ester **I** with nitryl chloride is that the tricycloheptane substrate has a considerably lower ionization potential as compared to sulfone **II**. Therefore, nitryl chloride can act here as both radical and electrophilic reagent [13].

## EXPERIMENTAL

The elemental compositions were determined on a Hewlett-Packard HP-185B CHN analyzer. The <sup>1</sup>H and <sup>13</sup>C NMR spectra were measured on a Bruker AC-300 spectrometer at 300.130 and 75.468 MHz, respectively, using CDCl<sub>3</sub> as solvent. The IR spectra were recorded in KBr on an InfraLYuM FT-02 spectrometer with Fourier transform. GLC analysis was performed on a Chrom-41 chromatograph equipped with a flame ionization detector and a glass column,

1200×3 mm, packed with 3% of OV-17 on Inerton N-Super (0.125–0.160 mm); carrier gas nitrogen, flow rate 40 ml/min; oven temperature 210°C, injector temperature 260°C. The components were quantitated using internal normalization technique; the calibrating factors for all compounds were assumed to be equal to unity. Analytical thin-layer chromatography was performed on Silufol UV-254 plates using hexane–diethyl ether (1:1) as eluent; development with iodine vapor. Aluminum oxide of activity grade II and silica gel L (40–100 μm) were used for column chromatography; eluent light petroleum ether–diethyl ether, (2–3):1. Tricycloheptanes **I** [14] and **II** [15] with a purity of no less than 97% were synthesized by known methods.

**Reaction of tricycloheptanes I and II with a mixture of dinitrogen tetraoxide and nitrosyl chloride (general procedure).** A mixture of anhydrous Pb(NO<sub>3</sub>)<sub>2</sub> and coarse quartz sand (1:2) was heated in a heat-resistant glass round-bottom flask using a gas burner. The released dinitrogen tetraoxide was passed through a 20×250-mm glass tube filled with anhydrous potassium chloride. The yellow–green gas consisting of NOCl and N<sub>2</sub>O<sub>4</sub> at a ratio of ~8:1 was collected in a receiver cooled to –15°C using an ice–salt bath. About 1 ml of the condensate was diluted with 5 ml of cold anhydrous diethyl ether, and a solution of 6 mmol of compound **I** or **II** in 5 ml of diethyl ether was added. The mixture was kept for 3 h at –5 to 0°C, and 10 ml of a saturated solution of sodium hydrogen carbonate was added with care. The organic layer was separated, the aqueous layer was extracted with diethyl ether (20×3 ml), the extracts were combined with the organic phase and dried over MgSO<sub>4</sub>, and the solvent was removed under reduced pressure (water-jet pump). The solid residue was analyzed by GLC and <sup>1</sup>H NMR. From ester **I** we obtained compound **III** as the only product which was purified by recrystallization. The product mixture obtained from sulfone **II** contained two compounds at a ratio of 10:1 (according to the <sup>1</sup>H NMR data). The major product, nitro chloride **IV**, was isolated by column chromatography on silica gel. We failed to isolate and identify the minor product.

**Methyl *exo*-6-chloro-*syn*-7-nitrobicyclo[3.1.1]-heptane-*endo*-6-carboxylate (III).** Yield 75%, mp 92–93°C (from diethyl ether), *R*<sub>f</sub> 0.73, *R*<sub>t</sub> 4.5 min (140°C). IR spectrum, *v*, cm<sup>-1</sup>: 542 m, 690 m, 1094 m, 1126 s, 1385 s (NO<sub>2</sub>, asym.), 1447 s, 1538 v.s (NO<sub>2</sub>, sym.), 1741 v.s (C=O). <sup>1</sup>H NMR spectrum, *δ*, ppm: 1.31–1.46 m (2H, 3-H), 2.20–2.42 m (4H, 2-H, 4-H), 3.31 br.d (2H, 1-H, 5-H, *J* = 5.5 Hz), 3.87 s (3H,

OMe), 5.19 t (1H, 7-H, *J* = 5.5 Hz). <sup>13</sup>C NMR spectrum, *δ*<sub>C</sub>, ppm: 11.4 (C<sup>3</sup>), 22.5 (C<sup>2</sup>, C<sup>4</sup>), 51.0 (C<sup>1</sup>, C<sup>5</sup>), 53.0 (OMe), 63.4 (C<sup>7</sup>), 75.2 (C<sup>6</sup>), 167.0 (C=O). Found, %: C 46.32; H 5.26; N 5.88. C<sub>9</sub>H<sub>12</sub>ClNO<sub>4</sub>. Calculated, %: C 46.27; H 5.18; N 5.59.

***exo*-6-Chloro-*syn*-7-nitrobicyclo[3.1.1]hept-*endo*-6-yl phenyl sulfone (IV).** Yield 56%, mp 158–159°C (from acetone–hexane), *R*<sub>t</sub> 8.7 min. IR spectrum, *v*, cm<sup>-1</sup>: 563 m, 617 m, 686 m, 721 m, 1153 v.s, 1315 v.s, 1385 s (NO<sub>2</sub>, asym.), 1446 s, 1527 v.s (NO<sub>2</sub>, sym.). <sup>1</sup>H NMR spectrum, *δ*, ppm: 1.31–1.52 m and 1.84–2.04 m (1H each, 3-H), 2.39–2.56 m and 2.92–3.09 m (2H each, 2-H, 4-H), 3.49 br.d (2H, 1-H, 5-H, *J* = 5.8 Hz), 5.11 t (1H, 7-H, *J* = 5.8 Hz), 7.63 t (2H, H<sub>arom</sub>, *J* = 7.3 Hz), 7.74 t (1H, H<sub>arom</sub>, *J* = 7.3 Hz), 7.95 d (2H, H<sub>arom</sub>, *J* = 7.3 Hz). <sup>13</sup>C NMR spectrum, *δ*<sub>C</sub>, ppm: 12.5 (C<sup>3</sup>), 23.8 (C<sup>2</sup>, C<sup>4</sup>), 54.3 (C<sup>1</sup>, C<sup>5</sup>), 75.3 (C<sup>7</sup>), 80.1 (C<sup>6</sup>); 129.1, 129.5, 134.5, 136.6 (C<sub>arom</sub>). Found, %: C 49.32; H 4.28; N 4.38. C<sub>13</sub>H<sub>14</sub>ClNO<sub>4</sub>S. Calculated, %: C 49.45; H 4.47; N 4.44.

**Reaction of tricycloheptane II with nitryl chloride.** A solution of 1.17 g of sulfone **II** in 20 ml of anhydrous chloroform containing 0.5 g of Na<sub>2</sub>CO<sub>3</sub> was cooled to –5°C, and a slight stream of NO<sub>2</sub>Cl {prepared by slowly adding 4.7 ml of chlorosulfonic acid to a mixture of 3 ml of nitric acid (*d* = 1.51 g/cm<sup>3</sup>) and 3 ml of 30% oleum according to the procedure described in [3]} was passed through the solution over a period of 30 min under stirring. The mixture was then stirred for an additional 1 h at 0°C, excess nitrosyl chloride was purged off with a slight stream of nitrogen (until the mixture turned light yellow), and the mixture was washed with a 5% solution of sodium carbonate and with water. The organic phase was dried over CaCl<sub>2</sub> and evaporated under slightly reduced pressure to obtain 1.49 g of a crystallizable oily material which was analyzed by TLC, GLC, and <sup>1</sup>H NMR. The product contained compounds **Va**, **Vb**, and **VI** at a ratio of 17:2.5:1. By column chromatography on aluminum oxide we isolated a mixture of dichlorides **Va** and **Vb** and cyclohexenyl sulfone **VI**. The subsequent recrystallizations gave 1.02 g (67%) of compound **Va** and 62 mg (4%) of **VI**. Compound **Vb**, 0.12 g (8%), was characterized by spectral methods as a sample with a stereochemical purity of 65%. Analogous reaction of tricycloheptane **II** with NO<sub>2</sub>Cl in diethyl ether gave a mixture of dichlorides **Va** and **Vb** at a ratio of 7.5:1 in an overall yield of 87%.

***endo*-6,*syn*-7-Dichlorobicyclo[3.1.1]hept-*exo*-6-yl phenyl sulfone (Va).** mp 161–162°C (from acetone–



hexane),  $R_f$  0.63,  $R_t$  6.0 min. IR spectrum,  $\nu$ ,  $\text{cm}^{-1}$ : 2970 w, 2937 w, 1452 m, 1324 s, 1307 s, 1151 v.s., 1090 m, 883 m, 692 m, 563 s.  $^1\text{H}$  NMR spectrum,  $\delta$ , ppm: 1.51–1.68 m and 1.68–1.86 (1H each, 3-H), 2.10–2.26 and 2.30–2.48 (2H each, 2-H, 4-H), 3.35 br.d (2H, 1-H, 5-H,  $J = 6.0$  Hz), 5.51 m (1H, 7-H,  $J = 6.0$  Hz), 7.60 t (2H,  $H_{\text{arom}}$ ,  $J = 7.5$  Hz), 7.73 t (1H,  $H_{\text{arom}}$ ,  $J = 7.5$  Hz), 7.99 d (2H,  $H_{\text{arom}}$ ,  $J = 7.5$  Hz).  $^{13}\text{C}$  NMR spectrum,  $\delta_c$ , ppm: 12.4 ( $\text{C}^3$ ), 25.7 ( $\text{C}^2$ ,  $\text{C}^4$ ), 49.1 ( $\text{C}^1$ ,  $\text{C}^5$ ), 54.5 ( $\text{C}^7$ ), 87.7 ( $\text{C}^6$ ); 128.9, 130.5, 134.4, 135.2 ( $\text{C}_{\text{arom}}$ ). Found, %: C 50.98; H 4.73.  $\text{C}_{13}\text{H}_{14}\text{Cl}_2\text{O}_2\text{S}$ . Calculated, %: C 51.16; H 4.62.

**exo-6, syn-7-Dichlorobicyclo[3.1.1]hept-endo-6-yl phenyl sulfone (Vb).**  $R_f$  0.63,  $R_t$  5.0 min.  $^1\text{H}$  NMR spectrum,  $\delta$ , ppm: 1.88–2.09 (2H, 3-H), 2.25–2.50 (2H, overlapped by the signal of Va) and 2.75–2.90 (2H) (2-H, 4-H), 3.11 br.d (2H, 1-H, 5-H,  $J = 5.7$  Hz), 5.01 t (1H, 7-H,  $J = 5.7$  Hz); signals from the aromatic protons were overlapped by those of isomer Va.  $^{13}\text{C}$  NMR spectrum,  $\delta_c$ , ppm: 13.2 ( $\text{C}^3$ ), 24.6 ( $\text{C}^2$ ,  $\text{C}^4$ ), 54.6 ( $\text{C}^1$ ,  $\text{C}^5$ ), 56.3 ( $\text{C}^7$ ), 84.4 ( $\text{C}^6$ ); 128.9, 129.4, 134.1, 137.0 ( $\text{C}_{\text{arom}}$ ). Found, %: C 50.89; H 4.78.  $\text{C}_{13}\text{H}_{14}\text{Cl}_2\text{O}_2\text{S}$ . Calculated, %: C 51.16; H 4.62.

**6-(Dichloromethyl)cyclohex-1-en-1-yl phenyl sulfone (VI).** mp 86–87°C (from acetone),  $R_f$  0.44,  $R_t$  6.6 min. IR spectrum,  $\nu$ ,  $\text{cm}^{-1}$ : 625 s, 686 s, 721 s, 756 m, 1087 m, 1145 v.s., 1315 s, 1446 s, 2920 w, 2963 w.  $^1\text{H}$  NMR spectrum,  $\delta$ , ppm: 1.48–1.61 m (1H), 1.76–1.89 m (1H), 2.04–2.19 m (1H), 2.27–2.44 m (3H), 3.09 br.s (1H, 6-H), 6.61 d (1H,  $\text{CHCl}_2$ ,  $J = 2.9$  Hz), 7.40 t (1H, 2-H,  $J = 4.4$  Hz), 7.58 t (2H,  $H_{\text{arom}}$ ,  $J = 7.3$  Hz), 7.66 t (1H,  $H_{\text{arom}}$ ,  $J = 7.3$  Hz), 7.88 d (2H,  $H_{\text{arom}}$ ,  $J = 7.3$  Hz).  $^{13}\text{C}$  NMR spectrum,  $\delta_c$ , ppm: 18.6, 22.6, 25.7, 42.6 ( $\text{C}^6$ ), 74.8 ( $\text{CHCl}_2$ ), 127.7, 129.4, 133.5 ( $\text{C}_{\text{arom}}$ ), 138.7 ( $\text{C}_{\text{arom}}$  or  $\text{C}^1$ ), 139.6 ( $\text{C}^1$  or  $\text{C}_{\text{arom}}$ ), 146.4 ( $\text{C}^2$ ). Found, %: C 51.22; H 4.73.  $\text{C}_{13}\text{H}_{14}\text{Cl}_2\text{O}_2\text{S}$ . Calculated, %: C 51.16; H 4.62.

**Reaction of sulfone II with (dichloro- $\lambda^3$ -iodanyl)-benzene.** A mixture of 0.5 g of sulfone II and 0.6 g of (dichloro- $\lambda^3$ -iodanyl)benzene in 15 ml of carbon tetrachloride was placed into a quartz tube, and the tube was tightly capped and irradiated with a DRT-400 lamp over a period of 9 h at 20°C. The precipitate of unreacted (dichloro- $\lambda^3$ -iodanyl)benzene was filtered off, the solvent was removed from the filtrate under slightly reduced pressure, and the solid residue was washed with 10 ml of diethyl ether to remove iodobenzene. We thus isolated 0.42 g (64%) of a mixture of dichlorides Va and Vb at a ratio of 9:1.

**Reaction of sulfone II with chlorine.** A solution of 0.5 g of sulfone II in 10 ml of carbon tetrachloride was added under stirring and cooling to 0°C to a saturated solution of chlorine in 50 ml of carbon tetrachloride. The mixture was stirred for 30 min (until the initial sulfone disappeared according to the TLC data). The solvent was removed under reduced pressure to obtain 0.67 g of a colorless oily material which contained (according to the GLC data), 55% of compound VI and 7 unidentified compounds with the following retention times, min: 1.9 (1%), 2.6 (3%), 3.4 (3%), 4.2 (5%), 8.4 (8%), 10.3 (12%), 12.5 (13%). By column chromatography on aluminum oxide we isolated 0.19 g (31%) of crystalline compound VI.

## REFERENCES

- Vasin, V.A., Kostryukov, S.G., Bolusheva, I.Yu., and Razin, V.V., *Russ. J. Org. Chem.*, 2008, vol. 44, p. 511.
- Bräuer, G., *Handbuch der präparativen anorganischen Chemie*, Stuttgart: Enke, 1981.
- Karyakin, Yu.V. and Angelov, I.I., *Chistye khimicheskie veshchestva* (Pure Chemicals), Moscow: Khimiya, 1974, p. 21.
- Brown, D.W., *J. Chem. Educ.*, 1985, vol. 62, p. 209.
- Wiberg, K.B. and Hess, B.A., *J. Org. Chem.*, 1966, vol. 31, p. 2250.
- O'Bannon, P.E. and Dailey, W.P., *Tetrahedron*, 1990, vol. 46, p. 7341.
- Kadzyauskas, P.P. and Zefirov, N.S., *Usp. Khim.*, 1968, vol. 37, p. 1243.
- Vasin, V.A., Kostryukov, S.G., and Razin, V.V., *Russ. J. Org. Chem.*, 1998, vol. 34, p. 1136.
- Borbulevych, O.Ya., Semenov, A.V., Vasin, V.A., and Razin, V.V., *Acta Crystallogr., Sect. E*, 2002, vol. 58, p. 925.
- Vasin, V.A., Bolusheva, I.Yu., Surmina, L.S., Buevich, A.V., Sergeev, N.M., Tanaseichuk, B.S., and Zefirov, N.S., *Zh. Org. Khim.*, 1990, vol. 26, p. 1501.
- Christl, M., *Advances in Strain in Organic Chemistry*, Greenwich: JAI, 1995, vol. 4, p. 163.
- Wiberg, K.B. and Szeimies, G., *J. Am. Chem. Soc.*, 1970, vol. 92, p. 571.
- Beger, J., *J. Prakt. Chem.*, 1967, vol. 35, p. 326; Zyk, N.V., Bondarenko, O.B., Nesterov, E.E., and Zefirov, N.S., *Russ. J. Org. Chem.*, 1999, vol. 35, p. 980.
- Razin, V.V., Eremenko, M.V., and Ogloblin, K.A., *Zh. Org. Khim.*, 1978, vol. 14, p. 973.
- Vasin, V.A., Bolusheva, I.Yu., Chernyaeva, L.V., Tanaseichuk, B.S., Surmina, L.S., and Zefirov, N.S., *Zh. Org. Khim.*, 1990, vol. 26, p. 1509.