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Reaction Products of Methyl Tricyclo[4.1.0.0^{2,7}]heptane-1-carboxylate and Tricyclo[4.1.0.0^{2,7}]hept-1-yl Phenyl Sulfone with Dinitrogen Tetraoxide

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Abstract—The reaction of methyl tricyclo[4.1.0.0^{2,7}]heptane-1-carboxylate with dinitrogen tetraoxide in diethyl ether at –10 to 0°C, followed by treatment of the reaction mixture with methanol, gave approximately equal amounts of methyl *exo*,*syn*-6,7-dinitro- and *exo*-6-hydroxy-*syn*-7-nitrobicyclo[3.1.1]heptane-*endo*-6-carboxylates. Tricyclo[4.1.0.0^{2,7}]hept-1-yl phenyl sulfone reacted with dinitrogen tetraoxide under analogous conditions to produce a mixture of diastereoisomeric *exo*,*syn*- and *endo*,*syn*-6,7-dinitro-6-phenylsulfonylbicyclo[3.1.1]heptanes and 6,6-dimethoxy-*endo*-7-nitrobicyclo[3.1.1]heptane at a ratio of 4.5:2:1. Probable factors responsible for the different stereoselectivities in the addition of N_2O_4 at the central C^1–C^7 bond of the initial tricycloheptane compounds were discussed. The structural parameters of the dinitro ester and related dinitro sulfone were compared on the basis of the X-ray diffraction data.

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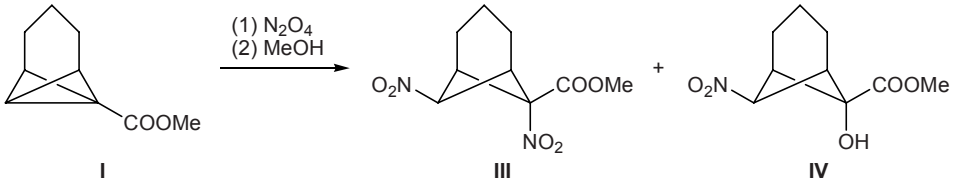
Addition reactions of dinitrogen tetraoxide to olefins provide a convenient synthetic route to nitro-substituted hydrocarbons [1]. The high strain energy of bicyclo[1.1.0]butane and olefin-like character of the central carbon–carbon bond therein [2] suggest that addition of N_2O_4 at the C^1–C^7 bond of bicyclo[1.1.0]-butane is possible. We previously [3] were the first to present experimental proofs for the above assumption: The reaction of methyl tricyclo[4.1.0.0^{2,7}]heptane-1-carboxylate (**I**) (which may be regarded as a bridged bicyclo[1.1.0]butane derivative) with dinitrogen tetraoxide gave dinitro compound **III** which was formed just via addition of N_2O_4 at the central bicyclobutane bond. The structure of **III** was proved by X-ray analysis [4]. Later on, Archibald et al. [5] reported on the synthesis of ethyl 1,3-dinitrocyclobutane-1-carboxylate by reaction of dinitrogen tetraoxide with ethyl bicyclo-[1.1.0]butane-1-carboxylate which is structurally related to ester **I**. The reaction of parent bicyclo[1.1.0]-butane with dinitrogen tetraoxide in diethyl ether at –78°C gave no nitrogen-containing compounds; instead, 3-ethoxycyclobutanone was obtained as the only product [6]. It was presumed [6] that dinitrogen tetraoxide in the reaction with bicyclo[1.1.0]butane acts as a source of electrophilic nitrosyl cation, leading to the formation of 3-ethoxycyclobutanone oxime which then undergoes hydrolysis to 3-ethoxycyclobutanone. These data led us to conclude that the presence of an electron-withdrawing substituent in the bridgehead position is likely to favor addition of N_2O_4 at the central bicyclobutane bond.

In the present communication we give more detailed data on the reaction of ester **I** with N_2O_4, as well as on the nitration of one more tricycloheptane compound having an electron-withdrawing group in the bridgehead position, sulfone **II**. The reactions were carried out using dinitrogen tetraoxide preliminarily dried by distillation over phosphoric anhydride [7].

The reaction of tricycloheptane **I** with excess liquid N_2O_4 was carried out in diethyl ether at –10°C. After treatment of the reaction mixture with methanol, we detected (GLC, NMR) norpinane derivatives **III** and **IV** at a ratio of ~1.1:1 (Scheme 1).

Compounds **III** and **IV** were isolated as individual substances by column chromatography on aluminum oxide, and their structure was confirmed by the IR and ^{1}H and ^{13}C NMR spectra. In particular, the configuration of substituent on C^7 in each of compounds **III** and

Scheme 1.



I III IV

IV is determined by the presence of a triplet signal from 7-H in the ^{1}H NMR spectrum [8, 9]. Taking into account that the structure of norpinane derivative **III** was unambiguously established previously by X-ray analysis, the *endo* orientation of the methoxycarbonyl group in molecule **IV** was assigned on the basis of the observed similarity in the chemical shifts of ^{1}H and ^{13}C in the trimethylene fragments of **III** and **IV**.

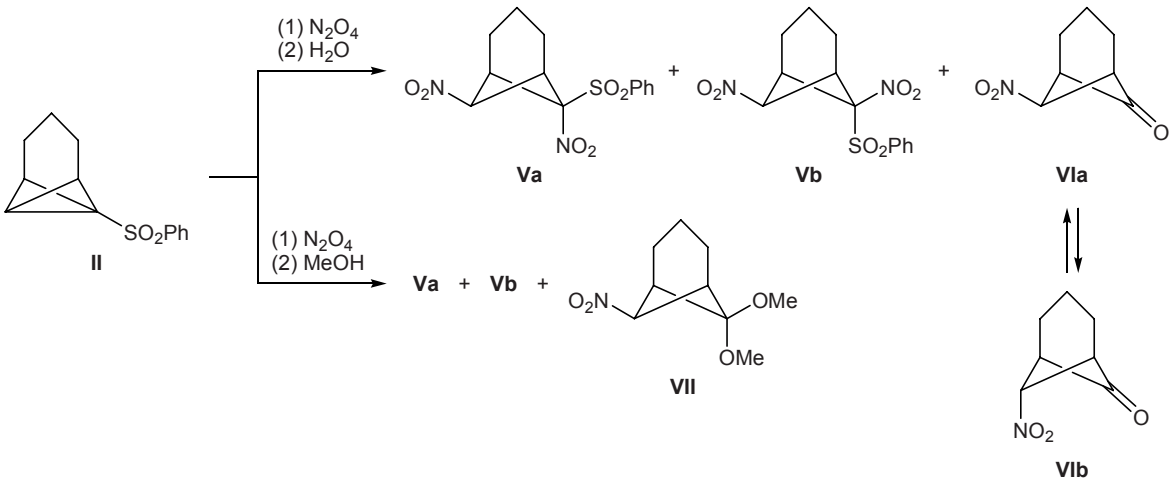
The reactions of sulfone **II** with liquid N_2O_4 were carried out at –10 to 0°C in diethyl ether or methylene chloride using excess reagent; the reaction mixtures were then treated with water or methanol. In the first case we obtained stereoisomeric dinitro sulfones **Va** and **Vb** and nitro ketone **VIa** at a ratio of 2.9:1:3.5 (according to the ^{1}H NMR data), while in the second case a mixture of dinitro sulfones **Va** and **Vb** and nitro dimethyl acetal **VII** at a ratio of 4.5:2:1 was formed (Scheme 2).

Compounds **Va**, **VIa**, and **VII** were isolated as individual substances by column chromatography on silica gel. The stereochemical purity of dinitro sulfone **Vb** was 90%. Chromatographic separation on aluminum oxide of the product mixture obtained after treatment of the reaction mixture with water gave dinitro sulfones **Va** and **Vb** and nitro ketone **VIb**. Presumably, the latter was formed by epimerization of **VIa** over aluminum oxide as catalyst [10].

The structure of compounds **V**–**VII** was confirmed by the IR and NMR spectra. The nitro groups in their molecules gave rise to two strong absorption bands in the IR spectra at ~1385 and ~1535 cm^{-1}; the bands at 1165 and 1335 cm^{-1} were assigned to the sulfonyl group. Ketones **VIa** and **VIb** characteristically displayed a strong absorption band at 1790 cm^{-1} due to stretching vibrations of the carbonyl group [11]. The configuration at C^7 unambiguously followed from the presence of a triplet signal from 7-H in the ^{1}H NMR spectra of nitro derivatives **Va**, **Vb**, **VIa**, and **VII** or a singlet from the same proton in the spectrum of **VIb** [8, 9]. The strong difference between the chemical shifts of 7-H in stereoisomeric dinitro sulfones **Va** and **Vb** allowed us to assign configuration at C^6 in their molecules with account taken of known stronger long-range deshielding effect of sulfo group as compared to nitro group [12]. The structure of dinitro sulfone **Va** was finally proved by X-ray analysis (see figure).

The dihedral angle between the $C^1C^2C^4C^5$ and $C^2C^3C^4$ planes in molecule **Va** is about 165°, and the C^3 atom deviates from the $C^1C^2C^4C^5$ plane by 0.211 Å toward C^7. The corresponding dihedral angle in the molecule of ester **III** [4] is equal to 178.2°, i.e., the trimethylene bridge is almost planar. Both nitro groups in **Va** are oriented almost orthogonally to the plane passing through the C^3, C^6, and C^7 atoms. The C^1–C^7

Scheme 2.



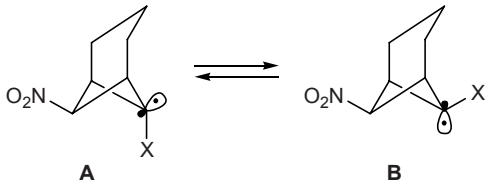
II Va Vb VIa VII VIb

and C^5–C^7 bonds (average length 1.537 Å) in the cyclobutane fragment of **Va** are shorter than the C^1–C^6 and C^5–C^6 bonds (average length 1.546 Å; cf. 1.536 and 1.553 Å, respectively, in molecule **III**); the dihedral angle between the $C^6C^1C^5$ and $C^7C^1C^5$ planes in **Va** is 136.4° (cf. 139.7° in **III**).

We believe that the addition of dinitrogen tetraoxide to bicyclobutane derivatives **I** and **II** follows a radical chain mechanism. As might be expected on the basis of published data [13], N-nitro radical generated by decomposition of N_2O_4 attacks the central C^1–C^7 bond in the substrate at the β-position with respect to the electron-withdrawing substituent with strict *endo* selectivity. As a result, the corresponding norpinanyl radical is formed. In the second chain propagation step dinitrogen tetraoxide acts as carrier of nitro and nitrosyl groups which are transferred to norpinanyl radical, yielding dinitronorpinane derivative and nitronorpinanyl nitrite [14]. The latter is converted into nitronorpinanol **IV** (in the reaction with ester **I**) or into nitronorpinanone **VIa** or its acetal **VII** (in the reaction with sulfone **II**) upon treatment of the reaction mixture with water or methanol.

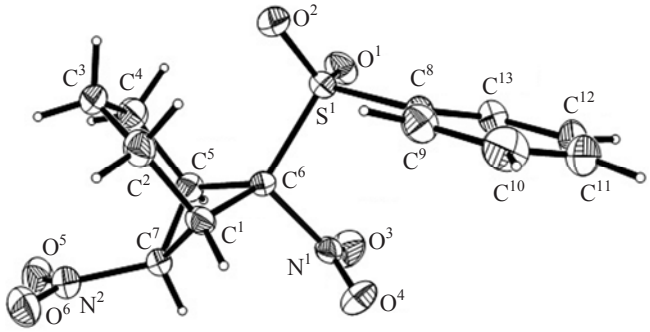
Special comments should be given to the observed difference in the stereochemistry of addition to tricycloheptane derivatives **I** and **II**. This difference is determined by stereoselectivity of the second step of the process, transfer of nitro or nitrosyloxy group to the reaction center in intermediate norpinan-6-yl radical, and is likely to be related to specific structure of the latter in each case. Initially, the intermediate always has conformation **A**, and it can undergo reversible isomerization into conformer **B** (Scheme 3).

Scheme 3.



X = CO_2Me, SO_2Ph.

Presumably, the *anti*-stereoselectivity of the nitration decreases in going from ester **I** to sulfone **II** due to increased barrier to inversion in intermediate **A** (X = SO_2Ph), which is stabilized by participation of *d*-orbitals on the sulfur atom. The inversion of radical **A** (X = CO_2Me) requires less energy, and radical **B** thus formed is more reactive due to the lack of steric shielding of the reaction center by the C^3H_2 fragment.



Structure of the molecule of *exo*-6,*syn*-7-dinitro-*endo*-6-phenylsulfonylbicyclo[3.1.1]heptane (**Va**) according to the X-ray diffraction data (one of the two crystallographically independent molecules is shown).

Analogous difference in the stereoselectivities of radical addition of benzenethiol and benzenesulfonyl bromide to the same tricycloheptane substrates (*anti* addition to ester **I** and *syn* addition to sulfone **II**) was reported previously [15, 16]; a similar patter was also typical of nucleophilic addition (e.g., of sodium methoxide and sodium methanethiolate) to compounds **I** and **II** [10, 17, 18].

EXPERIMENTAL

The elemental compositions were determined on an HP-185B CHN analyzer. The ^1H and ^{13}C NMR spectra were measured on a Bruker DPX-300 spectrometer at 300.130 and 75.468 MHz, respectively, from solutions in $CDCl_3$. The IR spectra were recorded in KBr on an InfraLYuM FT-02 instrument 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 140°C, injector temperature 210°C. The components were quantitated by the internal normalization technique; the calibration factors for all components were assumed to be equal to unity. Analytical thin-layer chromatography was performed using Silufol UV-254 plates (hexane–diethyl ether, 1 : 1; 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 : 1 to 3 : 1.

Compounds **I** [17] and **II** [19] were synthesized by known methods.

Reaction of methyl tricyclo[4.1.0.0^{2,7}]heptane-1-carboxylate (I) with dinitrogen tetraoxide. A solution of 0.73 g (4.8 mmol) of compound **I** in 10 ml of

anhydrous diethyl ether was cooled to –10°C, and a solution of 1.13 g (12.3 mmol) of dinitrogen tetraoxide (prepared according to [7] and purified by distillation over P_2O_5) in 10 ml of the same solvent was slowly added under stirring. The mixture was stirred for 30 min on cooling and treated with 5 ml of methanol. The solvent was removed under reduced pressure (water-jet pump) to obtain a greenish–yellow semicrystalline material. According to the ^{1}H NMR and GLC data, the product was a mixture of compounds **III** and **IV** at a ratio of 1.1:1 with a small impurity of two other compounds whose structure was not determined [presumably, substituted norpinanes, δ(*anti*-7-H) 5.07 and 5.19 ppm]. By column chromatography on aluminum oxide we isolated 0.62 g (52%) of dinitro ester **III** and 0.24 g (23%) of hydroxy nitro ester **IV**.

Methyl *exo*-6,*syn*-7-dinitrobicyclo[3.1.1]heptane-*endo*-6-carboxylate (III). mp 119–120°C (from CH_2Cl_2–hexane), R_f 0.58, R_t 8.1 min. IR spectrum, ν, cm^{-1}: 540 m, 665 m, 814 m, 1061 m, 1123 s, 1167 s, 1386 s (NO_2, asym.), 1456 s, 1553 v.s (NO_2, sym.), 1759 v.s (C=O). ^{1}H NMR spectrum, δ, ppm: 1.32–1.59 m (2H, 3-H), 2.45 br.t (4H, 2-H, 4-H, J = 7 Hz), 3.78 br.d (2H, 1-H, 5-H, J = 5.9 Hz), 3.87 s (3H, OMe), 4.89 t (1H, *anti*-7-H, J = 5.9 Hz). ^{13}C NMR spectrum, δ_C, ppm: 11.4 (C^3), 22.4 (C^2, C^4), 48.7 (C^1, C^5), 53.6 (OMe), 74.0 (C^7), 90.0 (C^6), 162.5 (C=O). Found, %: C 44.52; H 5.01; N 11.50. $C_9H_{12}N_2O_6$. Calculated, %: C 44.27; H 4.95; N 11.47.

Methyl *exo*-6-hydroxy-*syn*-7-nitrobicyclo[3.1.1]-heptane-*endo*-6-carboxylate (IV). mp 86–87°C (from CH_2Cl_2–hexane), R_f 0.21, R_t 6.2 min. IR spectrum, ν, cm^{-1}: 557 m, 814 m, 1060 m, 1385 s (NO_2, asym.), 1553 v.s (NO_2, sym.), 1734 br.s (C=O), 3449 m (OH). ^{1}H NMR spectrum, δ, ppm: 1.21–1.56 m (2H, 3-H), 2.25 br.t (4H, 2-H, 4-H, J = 7 Hz), 2.96 br.s (1H, OH), 3.12 br.d (2H, 1-H, 5-H, J = 5.9 Hz), 3.86 s (3H, OMe), 5.10 t (1H, *anti*-7-H, J = 5.9 Hz). ^{13}C NMR spectrum, δ_C, ppm: 12.1 (C^3), 22.3 (C^2, C^4), 50.0 (C^1, C^5), 52.4 (OMe), 74.3 (C^7), 76.5 (C^6), 169.9 (C=O). Found, %: C 50.12; H 6.14; N 6.37. $C_9H_{13}NO_5$. Calculated, %: C 50.23; H 6.09; N 6.51.

Reaction of phenyl tricyclo[4.1.0.0^{2,7}]hept-1-yl sulfone (II) with dinitrogen tetraoxide. *a*. A solution of 4.3 g (18.4 mmol) of compound **II** in 20 ml of methylene chloride was cooled to –15°C, and a solution of 7.45 g (81.5 mmol) of dinitrogen tetraoxide in 10 ml of the same solvent was slowly added under stirring. The mixture was stirred for 15 min and treated with 5 ml of methanol. The solvent was distilled off to obtain a semicrystalline material which contained compounds **Va**, **Vb**, and **VII** at a ratio of 4.5:2:1. By column chromatography on silica gel we isolated 1.92 g (32%) of dinitro sulfone **Va**, 0.72 g (12%) of dinitro sulfone **Vb**, and 0.18 g (5%) of nitro acetal **VII**.

b. The reaction was carried out as described above in *a*, but the mixture was treated with 3 ml of water. The organic layer was separated, the aqueous layer was extracted with methylene chloride (3×5 ml), the extracts were combined with the organic phase and dried over calcium chloride, and the solvent was removed. According to the ^{1}H NMR data, the residue contained compounds **Va**, **Vb**, and **VIa** at a ratio of 2.9:1:3.5. By column chromatography on silica gel we isolated 0.14 g (7%) of nitro ketone **VIa**, 1.74 g (29%) of dinitro sulfone **Va**, and 0.93 g (16%) of dinitro sulfone **Vb**. In an analogous experiment, chromatographic separation of the crude product in a column charged with aluminum oxide gave 0.11 g (5.2%) of nitro ketone **VIb**.

***exo*-6,*syn*-7-Dinitro-*endo*-6-phenylsulfonylbicyclo[3.1.1]heptane (Va).** mp 155–156°C (from MeOH), R_f 0.46. IR spectrum, ν, cm^{-1}: 555 m, 606 s, 687 m, 721 m, 760 m, 1165 s, 1335 s, 1450 m, 1551 v.s, 1553 v.s. ^{1}H NMR spectrum, δ, ppm: 1.36–1.51 m and 1.86–2.03 m (1H each, 3-H), 2.52–2.68 m and 2.92–3.08 m (2H each, 2-H, 4-H), 4.03 br.s (2H, 1-H, 5-H), 4.33 t (1H, *anti*-7-H, J = 5.5 Hz), 7.58–7.67 m (2H) and 7.75–7.87 (3H) (Ph). ^{13}C NMR spectrum, δ_C, ppm: 12.0 (C^3), 22.5 (C^2, C^4), 51.1 (C^1, C^5), 73.3 (C^7), 102.7 (C^6); 129.3, 129.6, 134.6, 135.6 (C_{arom}). Found, %: C 47.92; H 4.44; N 8.47. $C_{13}H_{14}N_2O_6S$. Calculated, %: C 47.85; H 4.32; N 8.58.

***endo*-6,*syn*-7-Dinitro-*exo*-6-phenylsulfonylbicyclo[3.1.1]heptane (Vb).** mp 173–178°C (from acetone–pentane), R_f 0.38, stereochemical purity 90%. ^{1}H NMR spectrum, δ, ppm: 1.25–1.40 m (2H, 3-H), 2.22–2.38 m and 2.42–2.56 m (2H each, 2-H, 4-H), 3.77 br.d (2H, 1-H, 5-H, J = 5.7 Hz), 5.49 t (1H, 7-H, J = 5.7 Hz), 7.59–7.72 m (2H) and 7.75–7.93 (3H) (Ph). ^{13}C NMR spectrum, δ_C, ppm: 10.3 (C^3), 22.4 (C^2, C^4), 48.1 (C^1, C^5), 70.6 (C^7), 100.6 (C^6); 129.6, 130.2, 135.6, 136.0 (C_{arom}). Found, %: C 47.90; H 4.53; N 8.68. $C_{13}H_{14}N_2O_6S$. Calculated, %: C 47.85; H 4.32; N 8.58.

6,6-Dimethoxy-*endo*-7-nitrobicyclo[3.1.1]heptane (VII). mp 68–69°C (from pentane), R_f 0.78. ^{1}H NMR spectrum, δ, ppm: 1.16–1.34 m and 1.47–1.64 m (1H each, 3-H), 1.96–2.10 m and 2.12–2.26 m (2H each, 2-H, 4-H), 3.10 br.s (2H, 1-H, 5-H), 3.24 s and 3.28 s (3H each, OMe), 4.55 t (1H, 7-H, J = 5.9 Hz). ^{13}C NMR spectrum, δ_C, ppm: 12.8 (C^3), 22.2

(C^2, C^4), 46.2 (C^1, C^5), 49.5 and 50.2 (OMe), 75.0 (C^7), 98.3 (C^6). Found, %: C 53.68; H 7.60; N 6.87. $C_9H_{15}NO_4$. Calculated, %: C 53.72; H 7.51; N 6.96.

***endo*-7-Nitrobicyclo[3.1.1]heptan-6-one (VIa).** mp 112–113°C (from hexane–diethyl ether), R_f 0.69. IR spectrum, ν, cm^{-1}: 459 w, 783 w, 806 w, 1389 m, 1451 m, 1536 v.s, 1539 s, 1782 s, 2886 w, 2951 m, 2978 m. ^{1}H NMR spectrum, δ, ppm: 1.52–1.79 m (2H, 3-H), 2.34–2.49 m and 2.65–2.81 m (2H each, 2-H, 4-H), 3.47 br.d (2H, 1-H, 5-H, J = 6.1 Hz), 4.95 t (1H, 7-H, J = 6.1 Hz). ^{13}C NMR spectrum, δ_C, ppm: 14.7 (C^3), 30.0 (C^2, C^4), 60.0 (C^1, C^5), 71.6 (C^7), 205.8 (C^6). Found, %: C 53.98; H 5.88; N 8.91. $C_7H_9NO_3$. Calculated, %: C 54.19; H 5.85; N 9.03.

***exo*-7-Nitrobicyclo[3.1.1]heptan-6-one (VIb).** mp 71–72°C (from hexane–diethyl ether), R_f 0.54. IR spectrum, ν, cm^{-1}: 721 w, 756 w, 1026 m, 1157 m, 1369 m, 1450 m, 1542 v.s, 1547 v.s, 1558 v.s, 1797 s. ^{1}H NMR spectrum, δ, ppm: 1.59–1.82 m (2H, 3-H), 2.53 br.t (4H, 2-H, 4-H, J = 5.8 Hz), 3.59 br.s (2H, 1-H, 5-H), 4.79 s (1H, 7-H). ^{13}C NMR spectrum, δ_C, ppm: 15.7 (C^3), 33.3 (C^2, C^4), 63.7 (C^1, C^5), 82.1 (C^7), 205.3 (C^6). Found, %: C 54.22; H 5.96; N 9.15. $C_7H_9NO_3$. Calculated, %: C 54.19; H 5.85; N 9.03.

X-Ray diffraction study of compound (Va). Transparent scaly single crystals, 0.50×0.35×0.20 mm. Total of 5039 reflections were measured at 293 K on a Siemens P3/PC automatic four-circle diffractometer (graphite monochromator, MoK_α irradiation, λ = 0.71073 Å, θ–2θ scanning, $2\theta_{max}$ = 50.12°, spherical segment $-1 \le h \le 13$, $0 \le k \le 23$, $0 \le l \le 31$). Averaging of equivalent reflections gave 4918 independent reflections (R_{int} = 0.0437), which were used in the structure solution and refinement. Rhombic crystals with the following unit cell parameters: a = 11.060(10), b = 19.444(16), c = 26.58(2) Å; V = 5716(9) Å^3; M 652.64; Z = 16; d_{calc} = 1.517 g/cm^3; μ = 0.259 mm^{-1}; F(000) = 2720; space group *Pbca*. The structure was solved by the direct method. All atoms were localized by successive syntheses of electron density. The refinement was performed with respect to F^2_{hkl} in anisotropic approximation for non-hydrogen atoms and isotropic approximation for hydrogen atoms. The final divergence factors were R_1 = 0.0659 [from 2785 reflections with $I > 2\sigma(I)$, F_{hkl}] and wR_2 = 0.1797 (from all 4918 reflections involved in the refinement procedure, F^2_{hkl}). Number of refined parameters 505, goodness of fit 1.019. The residual electron density from the Fourier difference series was 0.248 and –0.269 e Å^{-3}. No correction for absorption was introduced. All calculations were performed using SHELXTL ver. 5.1 software package [20]. A unit cell contained two crystallographically independent molecules with similar structures. The complete set of crystallographic data for compound **Va** was deposited to the Cambridge Crystallographic Data Center (entry no. CCDC 295617).

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