Synthesis and conformation of 6,8,10-trinitro-1,4-dioxaspiro[4.5]decane

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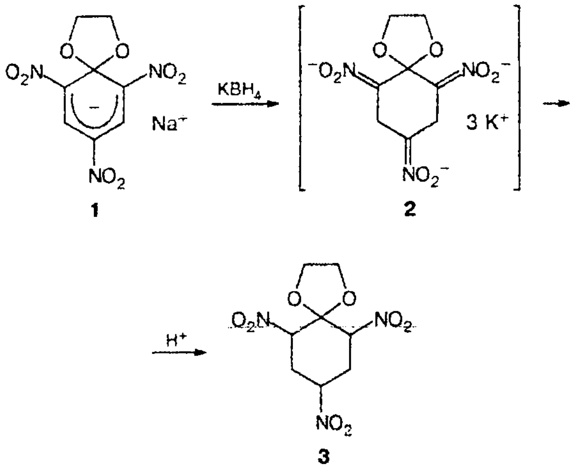
cis,trans-6,8,10-Trinitro-1,4-dioxaspiro[4.5]decane was synthesized by reduction of the corresponding spirocyclic anionic σ-complex with potassium tetrahydroborate. The molecular structure and the conformation of the title compound were established by X-ray diffraction analysis and ^{1}H NMR spectroscopy.

Key words: anionic σ-complexes, sodium 6,8,10-trinitro-1,4-dioxaspiro[4.5]deca-6,9-dienide, 6,8,10-trinitro-1,4-dioxaspiro[4.5]decane, X-ray diffraction study, molecular structure.

The reactions of nitrobenzenes with sodium tetrahydroborate yield different products depending on the structure of the substrate and the reaction conditions. In most cases, amines and products of incomplete reduction of the nitro group are formed.[1] From the synthetic standpoint, it is of interest to elucidate the possibility of selective reduction of nitroarenes under the action of $NaBH_4$ to form cycloaliphatic nitro compounds,[2] which attract attention of researchers as very valuable intermediates in various fields of organic synthesis.[3] Reduction of *symm*-trinitrobenzene derivatives containing substituents, such as a halogen atom, an alkoxy group, or the hydroxy group, with sodium tetrahydroborate afforded 1,3,5-trinitrocyclohexane,[4] *i.e.*, reduction of the aromatic ring was accompanied by hydrogenolysis of the above-mentioned substituents. Unlike the behavior of the above-mentioned aromatic derivatives, reduction of the Meisenheimer σ-complex, which was prepared from 2,4,6-trinitroanisole and potassium methoxide, with $NaBH_4$ was not accompanied by reduction of the methoxy groups and allowed the synthesis of 1,1-dimethoxy-2,4,6-trinitrocyclohexane.[5] In this connection, we studied the reaction of KBH_4 with the spirocyclic anionic σ-complex, *viz.*, with sodium 6,8,10-trinitro-1,4-dioxaspiro[4.5]deca-6,9-dienide (**1**).

It was found that the spirocyclic structure was retained upon reduction of adduct **1** (Scheme 1) to form 6,8,10-trinitro-1,4-dioxaspiro[4.5]decane (**3**). Previously, we detected the sodium salt of 1,3,5-tris(*aci*-nitro)cyclohexane as an intermediate in the reduction of 1,3,5-trinitrobenzene with sodium tetrahydroborate to trinitrocyclohexane.[6] It can be suggested that the reaction under study also gave a salt of 6,8,10-tris(*aci*-nitro)-1,4-dioxaspiro[4.5]decane (**2**).

Scheme 1



The IR spectra of the resulting compound **3** have absorption bands of the C—H bonds (ν(C—H) 3024,

Translated from *Izvestiya Akademii Nauk. Seriya Khimicheskaya*, No. 9, pp. 1713—1717, September, 1999.

Table 1. Results of resolution of the absorption IR spectra of compound **3** into components of the Gaussian (I), Lorentz (II), and superposition (III) forms

Vibration	Number of the peak	ν_{max}/cm^{-1}			I_{rel}			$D_{\nu_{1/2}}$/cm^{-1}		
		I	II	III	I	II	III	I	II	III
$\nu_{as}(NO_2)$	1	1516	1510	1510	2.2	1	1	34	3	4
		(1.95173)	(0.206061)	(0.181561)	(0.83365)	(1.96849)	(1.42017)	(2.81243)	(0.64157)	(0.43228)
	2	1526	1525	1524	1	1.2	1.3	10	9	9
		(0.245305)	(0.233477)	(0.238057)	(2.26495)	(1.42955)	(1.48693)	(1.01651)	(1.02359)	(0.980139)
	3	1544	1544	1544	2.7	1.9	2.1	16	9	8
		(0.250256)	(0.142879)	(0.148538)	(1.69811)	(1.39619)	(1.23642)	(0.696507)	(0.581992)	(0.874436)
	4	1559	1560	1560	1.9	1.4	1.8	11	8	10
		(0.247073)	(0.226591)	(0.258443)	(4.85654)	(1.8155)	(1.35577)	(0.893075)	(0.994599)	(0.874436)
	5	1572	1573	1575	2.2	1.2	1	22	25	16
		(0.669181)	(0.891326)	(0.570926)	(2.43872)	(1.70363)	(0.951786)	(3.71134)	(3.22948)	(1.40939)
$\nu_s(NO_2)$	1	1306	1305	1306	1.4	2.8	2.4	11	7	9
		(0.109282)	(0.051956)	(0.089839)	(0.190371)	(0.164277)	(0.539543)	(0.343926)	(0.17118)	(0.306032)
	2	1321	1323	1322	1.3	3	2.2	14	12	12
		(0.571075)	(0.122479)	(0.219622)	(2.30934)	(0.382255)	(1.17611)	(1.02796)	(0.47213)	(0.699064)
	3	1336	1336	1336	2.1	3.8	3.6	21	18	21
		(0.675053)	(0.160818)	(0.369587)	(0.453444)	(0.25048)	(0.666269)	(2.64844)	(0.718088)	(1.40373)
	4	1352	1352	1352	1	1	1	11	9	9
		(0.399568)	(0.208019)	(0.346911)	(2.15946)	(0.276108)	(1.25057)	(1.05553)	(1.04658)	(1.28129)
	5	1366	1367	1365	2.6	4.8	1.6	16	19	10
		(1.29892)	(0.187665)	(0.287908)	(3.34565)	(0.812856)	(2.24769)	(2.66233)	(0.816286)	(1.99469)
	6	1380	1382	1376	2.1	3.5	4.4	16	22	32
		(1.49632)	(0.332046)	(1.0175)	(3.7255)	(0.724768)	(1.01038)	(2.96312)	(1.60789)	(2.8226)
	7	1397	1397	1398	2.2	3.8	2.8	16	11	13
		(0.38418)	(0.068856)	(0.096051)	(0.693415)	(0.376734)	(0.894897)	(0.393376)	(0.301083)	(0.397575)
$\nu_{as}(COC)$	1	1108	1107	1107	1	1	1	23	13	20
		(0.656856)	(0.581691)	(0.507231)	(0.309476)	(2.29032)	(1.37816)	(2.58754)	(1.66792)	(5.00982)
	2	1121	1122	1121	1	1.6	1.1	8	12	9
		(1.02517)	(0.178768)	(0.293477)	(5.2952)	(1.07855)	(2.56358)	(1.39984)	(0.753562)	(0.706862)
	3	1129	1130	1129	1.3	1.7	1.2	9	13	11
		(0.781303)	(0.162341)	(0.253798)	(4.85847)	(1.02714)	(2.05796)	(2.41862)	(1.02906)	(1.06639)
	4	1138	1139	1139	1.2	1.2	1.1	12	9	12
		(1.10486)	(0.110687)	(0.38814)	(2.66592)	(0.013946)	(1.70769)	(2.00829)	(0.422724)	(0.739963)
$\nu_s(COC)$	1	1073	1075	1075	1	1	1	13	9	12
		(3.3372)	(0.21156)	(0.767834)	(3.61686)	(0.430468)	(2.14591)	(3.18239)	(0.64287)	(0.950376)
	2	1084	1084	1084	2.1	2.5	2	13	12	13
		(1.56161)	(0.12639)	(0.351018)	(3.04563)	(0.372545)	(3.98342)	(2.33679)	(0.567547)	(3.28686)

Note. Standard deviations are given in parentheses.

2982, and 2918 cm^{-1}), the nitro groups ($\nu_{as}(NO_2)$ 1542 cm^{-1} and $\nu_s(NO_2)$ 1370 cm^{-1}), and the C—O—C bond of the spiro ring (ν_{as}(C—O—C) 1128 cm^{-1} and ν_s(C—O—C) 1026 cm^{-1}). The absorption bands belonging to the nitro groups and the spiro ring have complex contours and consist of a number of substantially overlapping components, which is indicative of the nonequivalence of the nitro groups and of distortion of the symmetry of the spiro ring. The resolution of the observed contours into components, which are described by the Gaussian function, Lorentz function, or their superposition, demonstrates that the $\nu_{as}(NO_2)$, $\nu_s(NO_2)$, ν_{as}(C—O—C), and ν_s(C—O—C) bands consist of five, seven, four, and two components, respectively. Their spectral characteristics are given in Table 1. The best agreement between the calculated and experimental contours was obtained with the use of the Lorentz function.

The character of the ^{1}H NMR spectrum (400 MHz) of compound **3** in benzene (Fig. 1) indicates that **3** was obtained in the individual state. This is evidenced by the groups of well resolved signals for the protons of the ring, *viz.*, five multiplets (*A*, *B*, *C*, *G*, and *H*; see Fig. 1) with unit intensities and one signal with double intensity (*I* and *J*). The signals *D*, *E*, and *F* correspond to the four protons of the spiro ring. Three low-field signals (*A*, *B*, and *C*) belong to the methine protons. The width of the multiplet and the corresponding chemical shifts allow the determination of the orientation of the protons in the ring. Thus, the signals *A* (at δ 4.685) and *B* (δ 4.280) (Table 2) correspond to the protons in

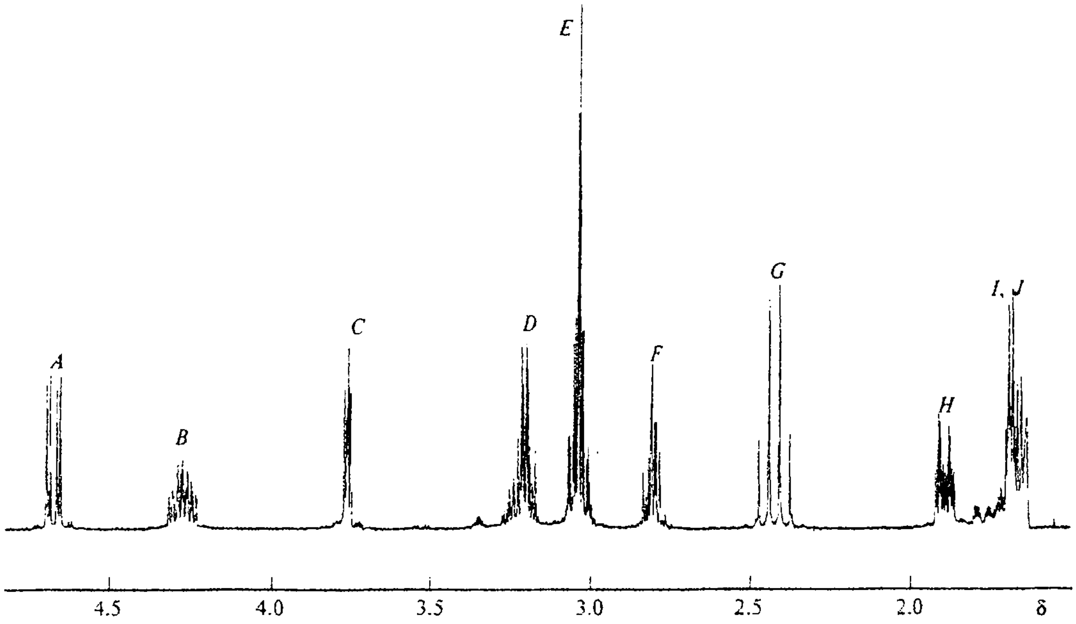


Fig. 1. ^{1}H NMR spectrum (400 MHz) of compound **3** in benzene-d$_6$.

Table 2. Experimental chemical shifts (δ) and spin-spin coupling constants (J)^a in the ^{1}H NMR spectra of 6,8,10-trinitro-1,4-dioxaspiro[4.5]decane (a solution in C_6D_6, 300 K)

Proton^b	δ	Protons	$J_{H,H}$/Hz
$H(2)_a$ (*A*)	4.685	$H(2)_a$, $H(3)_a$	12.70 (0.02)
$H(3)_a$ (*G*)	2.429	$H(2)_a$, $H(3)_e$	4.27 (0.02)
$H(3)_e$ (*H*)	1.899	$H(3)_a$, $H(3)_e$	−12.58 (0.02)
$H(4)_a$ (*B*)	4.280	$H(3)_a$, $H(4)_a$	12.51 (0.02)
$H(5)_a$ (*I*)	1.676	$H(3)_e$, $H(4)_a$	4.39 (0.02)
$H(5)_e$ (*J*)	1.708	$H(3)_e$, $H(5)_e$	2.22 (0.02)
$H(6)_e$ (*C*)	3.768	$H(4)_a$, $H(5)_a$	11.74 (0.03)
		$H(4)_a$, $H(5)_e$	4.98 (0.02)
		$H(5)_a$, $H(5)_e$	−12.31 (0.02)
		$H(5)_a$, $H(6)_e$	4.96 (0.03)
		$H(5)_e$, $H(6)_e$	2.77 (0.02)

[a] The spin-spin coupling constants were calculated within the framework of the seven-spin *ABCGHIJ* system; the rms deviation for 253 assigned theoretical transitions is 0.116 Hz. The standard deviations for the spin-spin coupling constants are given in parentheses. The long-range spin-spin coupling constants, which are no more than 0.5 Hz in magnitude (presumably), are not listed in the table.

[b] The assignment of the signals in the ^{1}H NMR spectrum (see Fig. 1) is given in parentheses.

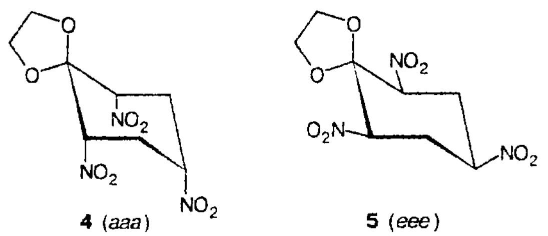
the axial orientation. In addition, a substantial width of the multiplet *B* enables one to unambiguously assign it to the $H(4)_a$ proton. The higher-field signal *C* (at δ 3.768) is characterized by low values of the vicinal spin-spin coupling constants (~5.0 and 3.0 Hz), which is unambiguously indicative of equatorial arrangement of the corresponding proton. The characteristic feature of this spectrum is the fact that the signal *G* belonging to one of the methylene protons appears as a regular quadruplet due to the accidentally equal values of one geminal and two vicinal constants. This situation can occur only if the signal *G* corresponds to the $H(3)_a$ proton. In this case, the signal *H* should belong to the $H(3)_e$ proton. The signals *I* and *J* correspond to the $H(5)_a$ and $H(5)_e$ protons, respectively, whose chemical shifts have close values due to the symmetrical arrangement of the nitro groups. It is impossible to determine the orientations of these two protons by qualitative consideration.

With the aim of unambiguously determining the conformation of compound **3**, which can exist as one of three stereoisomers, *viz.*, *cis,cis*, *cis,trans*, or *trans,trans*, each being represented by two conformers (Scheme 2, the *a* and *e* symbols denote the orientations of the nitro groups), complete analysis of the protons of the ring in the ^{1}H NMR spectrum was carried out with the use of the PAREMUS-LAOCOON program package.[7] The spectrum was analyzed within the framework of the seven-spin *ABCGHIJ* system (see Table 2). The initial values of the chemical shifts and the spin-spin coupling constants were estimated on the assumption that the spectrum under consideration is nearly a first-order spectrum. With the aim of revealing the orientations of the *I* and *J* protons, model calculations were carried out. Good visual agreement between the experimental and model spectra was observed only if the signals *I* and *J* were assigned to the $H(5)_a$ and $H(5)_e$ protons, respectively. The calculations were performed with the use of 62 spectral components assigned to 253 theoretical transitions.

The determined values of the spin-spin coupling constants unambiguously indicate that compound **3** is the *cis,trans* stereoisomer existing as conformer **8**.

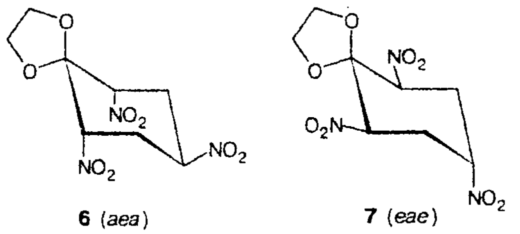
Scheme 2

cis,cis isomer:



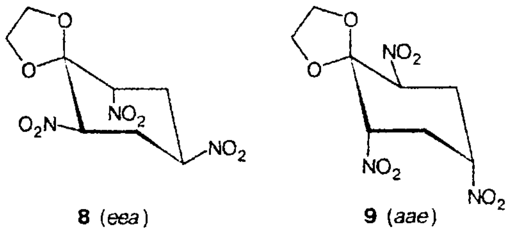
4 (*aaa*) **5** (*eee*)

trans,trans isomer:



6 (*aea*) **7** (*eae*)

cis,trans isomer:



8 (*eea*) **9** (*aae*)

The structure of compound **3** was confirmed by X-ray diffraction analysis (Fig. 2, Tables 3—5). The cyclohexane ring adopts a chair conformation. The C(1) and C(4) atoms deviate from the mean plane passing through the remaining atoms of the ring by −0.62 and 0.68 Å, respectively. The five-membered ring adopts an envelope conformation. The O(2) atom deviates from the C(1)O(1)C(7)C(8) plane by 0.30 Å. The substantially shortened C(7)—C(8) bond (1.443(7) Å) is apparently characterized by an averaged value due to significant thermal vibrations of the atoms. The nitro group at the C(2) atom is in the axial orientation (the C(4)—C(3)—C(2)—N(1) torsion angle is −75.5(3)°). The nitro groups at the C(4) and C(6) atoms are in equatorial orientations (the C(6)—C(5)—C(4)—N(2) and C(4)—C(5)—C(6)—N(3) torsion angles are −176.6(2)° and 178.9(2)°, respectively). The molecular packing in the crystal is determined by normal van der Waals contacts and has no specific features.

Hence, the most favorable conformation of compound **3** both in the crystalline phase and in solution is that with the diequatorial-axial arrangement of the nitro groups.

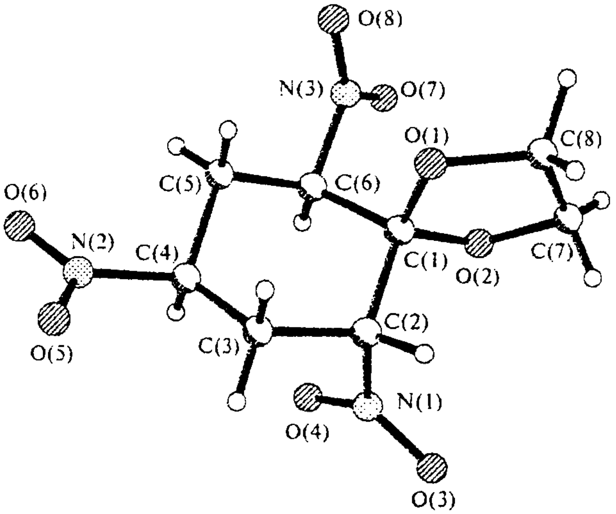


Fig. 2. Overall view of molecule **3**.

Table 3. Coordinates of nonhydrogen atoms ($\times 10^4$) and hydrogen atoms ($\times 10^3$) and equivalent isotropic thermal parameters ($U_{eq} \times 10^3$) in the structure of **3**

Atom	x	y	z	U_{eq}/Å^2
N(1)	5314(2)	1618(3)	1054(2)	50(1)
N(2)	2053(2)	853(3)	2853(2)	44(1)
N(3)	6188(2)	2980(3)	4729(2)	54(1)
O(1)	6544(2)	239(2)	3936(2)	60(1)
O(2)	7291(2)	1931(2)	2913(2)	59(1)
O(3)	5895(4)	1286(3)	331(2)	112(1)
O(4)	4729(2)	2696(3)	1039(2)	64(1)
O(5)	1648(2)	−250(3)	2463(3)	84(1)
O(6)	1456(2)	1644(3)	3341(2)	81(1)
O(7)	6848(3)	3980(3)	4664(2)	106(1)
O(8)	6154(2)	2386(3)	5614(2)	86(1)
C(1)	6180(2)	1311(3)	3145(2)	39(1)
C(2)	5373(2)	632(3)	2051(2)	41(1)
C(3)	4032(2)	195(3)	2178(2)	40(1)
C(4)	3354(2)	1354(3)	2692(2)	31(1)
C(5)	4128(2)	1846(3)	3836(2)	35(1)
C(6)	5393(2)	2421(3)	3626(2)	34(1)
C(7)	8297(4)	897(7)	3238(4)	89(2)
C(8)	7870(4)	−64(6)	4013(5)	94(1)
H(2)	587(2)	−188(3)	1885(2)	37(6)
H(3A)	355(3)	−110(3)	1458(2)	53(8)
H(3B)	417(3)	−548(3)	2693(2)	42(7)
H(4)	316(2)	2140(3)	2227(2)	32(6)
H(5A)	370(2)	2614(3)	4156(2)	41(7)
H(5B)	430(2)	1065(3)	4350(2)	37(7)
H(6)	526(2)	3170(3)	3166(2)	33(6)
H(7A)	896(4)	1467(4)	3398(4)	91(13)
H(7B)	836(5)	508(6)	2568(5)	138(20)
H(8A)	798(7)	−998(7)	3853(6)	179(28)
H(8B)	842(7)	54(8)	4829(6)	200(29)

Experimental

The IR spectra were recorded on a Specord IR-75 spectrometer in KBr pellets and Nujol mulls as well as in crystalline

Table 4. Bond lengths (*d*) in the structure of **3**

Bond	*d*/Å	Bond	*d*/Å
N(1)—O(4)	1.202(3)	O(1)—C(8)	1.423(4)
N(1)—O(3)	1.203(3)	O(2)—C(1)	1.399(3)
N(1)—C(2)	1.512(4)	O(2)—C(7)	1.449(5)
N(2)—O(5)	1.197(3)	C(1)—C(6)	1.534(3)
N(2)—O(6)	1.211(3)	C(1)—C(2)	1.553(4)
N(2)—C(4)	1.512(3)	C(2)—C(3)	1.521(4)
N(3)—O(7)	1.198(3)	C(3)—C(4)	1.518(3)
N(3)—O(8)	1.209(3)	C(4)—C(5)	1.520(3)
N(3)—C(6)	1.511(3)	C(5)—C(6)	1.520(3)
O(1)—C(1)	1.395(3)	C(7)—C(8)	1.443(7)

Table 5. Bond angles (ω) in the structure of **3**

Angle	ω/deg	Angle	ω/deg
O(4)—N(1)—O(3)	123.7(3)	O(2)—C(1)—C(2)	110.7(2)
O(4)—N(1)—C(2)	119.0(2)	C(6)—C(1)—C(2)	110.9(2)
O(3)—N(1)—C(2)	117.3(3)	N(1)—C(2)—C(3)	111.2(2)
O(5)—N(2)—O(6)	123.8(2)	N(1)—C(2)—C(1)	109.3(2)
O(5)—N(2)—C(4)	119.6(2)	C(3)—C(2)—C(1)	113.4(2)
O(6)—N(2)—C(4)	116.5(2)	C(4)—C(3)—C(2)	111.8(2)
O(7)—N(3)—O(8)	123.6(3)	N(2)—C(4)—C(3)	110.1(2)
O(7)—N(3)—C(6)	117.1(3)	N(2)—C(4)—C(5)	108.9(2)
O(8)—N(3)—C(6)	119.2(2)	C(3)—C(4)—C(5)	112.1(2)
C(1)—O(1)—C(8)	109.1(3)	C(4)—C(5)—C(6)	107.3(2)
C(1)—O(2)—C(7)	105.3(3)	N(3)—C(6)—C(5)	109.8(2)
O(1)—C(1)—O(2)	108.5(2)	N(3)—C(6)—C(1)	108.8(2)
O(1)—C(1)—C(6)	110.5(2)	C(5)—C(6)—C(1)	112.3(2)
O(2)—C(1)—C(6)	109.1(2)	C(8)—C(7)—O(2)	107.1(3)
O(1)—C(1)—C(2)	107.1(2)	O(1)—C(8)—C(7)	105.4(3)

films prepared from acetone solution. The differentiation of the experimental curve and resolution of the spectrum into elementary components were carried out using the BONEM GRAMS/386 program (version 2.03).

The ^{1}H NMR spectra of solutions in benzene-d$_6$ were recorded on a Varian VXR-400 spectrometer. The ^{1}H NMR spectrum was calculated with the use of the PAREMUS-LAOCOON program package[7] on an IBM PC computer.

***cis,trans*-6,8,10-Trinitro-1,4-dioxaspiro[4.5]decane (3).** A solution of KBH_4 (0.86 g, 16.0 mmol) in 50% aqueous methanol (20 mL) was added dropwise with stirring and cooling to 0—5 °C to a suspension of σ-complex **1** (1.0 g, 3.2 mmol), which was prepared according to a procedure reported previously,[8] in THF (20 mL). At the end of the reaction, the colored solution grew light and an intermediate precipitated. The precipitate was separated by decantation and dissolved in cold water (50 mL). A 20% solution of tartaric acid was gradually added with stirring and cooling to the resulting solution until the pH became 5.0, after which the pH was maintained for 3—4 h and then brought to 4.0. The reaction mixture was kept in a refrigerator for 15 h. The product was extracted with diethyl ether (3×50 mL), the extract was dried over $CaCl_2$, and the solvent was distilled off. The residue was dissolved in hot ethanol (10 mL), decolorized with activated carbon, filtered off, and slowly concentrated in air. Compound **3** was obtained as colorless crystals in 64% yield, m.p. 144—146 °C. Found (%): C, 34.60, 34.63; H, 4.01, 3.96; N, 15.14, 15.16. $C_8H_{11}N_3O_8$. Calculated (%): C, 34.66; H, 4.00; N, 15.16.

X-ray diffraction study of compound 3. Crystals of **3** ($C_8H_{11}N_3O_8$) are monoclinic. At 20 °C, a = 10.626(4) Å, b = 9.558(3) Å, c = 11.966(7) Å, β = 101.79(4)°, V = 1189.7(9) Å^3, d_{calc} = 1.348 g cm^{-3}, space group $P2_1/n$, Z = 4. The unit cell parameters and intensities of 3474 independent reflections (R_{int} = 0.021) were measured on an automated Siemens P3/PC diffractometer (Mo-Kα radiation, graphite monochromator, θ/2θ scanning technique, $2\theta_{max}$ = 60°). The structure was solved by the direct method with the use of the SHELXTL PLUS program package.[9] The positions of the hydrogen atoms were located from the difference electron density synthesis. The structure was refined by the full-matrix least-squares method with anisotorpic (isotropic for hydrogen atoms) thermal parameters based on F^2 using 2015 reflections to wR_2 = 0.135 (R_1 = 0.054 using 1449 reflections with $F > 4\sigma(F)$, s = 1.05). The atomic coordinates are given in Table 3. The bond lengths and bond angles are listed in Tables 4 and 5, respectively.

This work was financially supported by the Russian Foundation for Basic Research (Project Nos. 97-03-33783a and 96-15-97367).

References

1. M. Hudlicky, *Reduction in Organic Chemistry*, Ellis Horwood, Chichester, 1984, 309 pp.
2. T. Severin and R. Schmitz, *Chem. Ber.*, 1962, **95**, 1417.
3. G. A. Shvekhgeimer, V. I. Zvolinskii, and K. I. Kobrakov, *Khim. Geterotsikl. Soedin.*, 1986, 435 [*Chem. Heterocycl. Compd.*, 1986 (Engl. Transl.)].
4. L. A. Kaplan, *J. Am. Chem. Soc.*, 1964, **86**, 740.
5. V. A. Sokolova, M. D. Boldyrev, and B. V. Gidaspov, *Zh. Org. Khim.*, 1972, **8**, 770 [*J. Org. Chem. USSR*, 1972, **8** (Engl. Transl.)].
6. Yu. M. Atroshchenko, S. N. Nasonov, S. S. Gitis, A. Ya. Kaminsky, A. I. Mel'nikov, and I. V. Shakhkel'dyan, *Zh. Org. Khim.*, 1994, **30**, 632 [*Russ. J. Org. Chem.*, 1994, **30** (Engl. Transl.)].
7. S. S. Golotvin and V. A. Chertkov, *Izv. Akad. Nauk, Ser. Khim.*, 1997, 444 [*Russ. Chem. Bull.*, 1997, **46**, 423 (Engl. Transl.)].
8. T. I. Morozova, S. S. Gitis, A. Ya. Kaminsky, A. I. Glaz, A. I. Mel'nikov, and Yu. D. Grudtsyn, *Zh. Org. Khim.*, 1997, **33**, 1917 [*Russ. J. Org. Chem.*, 1997, **33** (Engl. Transl.)].
9. G. M. Sheldrick, *SHELXTL PLUS. PC Version, A System of Computer Programs for the Determination of Crystal Structure from X-ray Diffraction Data, Rev. 5.02*, Siemens Analytical X-ray Instruments Inc., Göttingen (Germany), 1994.

Received December 8, 1998