

Synthesis and characterization of 2,7-dihydro-1*H*-dinaphtho[*c,e*]tellurepin: a new heterocyclic telluride

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Abstract

Synthesis of the racemic cyclic telluride, i.e., 2,7-dihydro-1*H*-dinaphtho-*[c,e]*tellurepin (**1**), possessing a C₂ axis was based on the reaction of 2,2'-bis(bromomethyl)-1,1'-binaphthalene with potassium tellurocyanate in dry DMSO. Reaction of halogens with **1** gave the diiodo (**2**), dibromo (**3**) and dichloro (**4**) derivatives. Treatment of **1** with iodomethane and iodoethane gave the methyl- and ethyl tellurepinium iodides, **5** and **6**, respectively.

Compound **1** reduced the carbonyl groups in DDQ and TCQ to hydroxyl groups. Mononuclear palladium(II) complex, [(C₂₂H₁₆Te)₂PdCl₂], was prepared by reaction of **1** with [PdCl₂(NCPPh)₂].

All new compounds were characterized by elemental analysis and spectroscopic techniques.

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1. Introduction

There are a few publications [1–4] dealing with the synthesis of tellurium seven-membered ring compounds in contrast to a large amounts of research was concerned with five- and six-member rings [5,6]. The first report [1] described the synthesis of a steroidal telluacycloheptane from the reaction of a steroidal methanesulphonate with sodium telluride; yield and physical data of the product were not given [1]. The second paper [2] deals with the synthesis of 3*H*-benzotellurepin by reacting *o*-diethynylbenzene with tellurium powder, hydrazine and sodium hydroxide [2]. Our recent works [3,4] described the synthesis of 2,7-dihydro-1*H*-dibenzo[*c,e*]tellurepin either by reduction of 1,1-diiodo-2,7-dihydro-1*H*-dibenzo[*c,e*]tellurepin [3] or by reaction of 2,2'-bis(bromomethyl)biphenyl with potassium tellurocyanate [4]. Thus, as a part of research on the synthesis and donor properties of heterocyclic tellurium compounds [3,4,7,8], we

now report the synthesis of an axially dissymmetric cyclic telluride (i.e., 2,7-dihydro-1*H*-dinaphtho-*[c,e]*tellurepin (**1**)) in a chiral form. Furthermore, we report the reaction of this new cyclic telluride with halogens, alkyl iodides, quinones and palladium(II) complex.

2. Experimental

2.1. Synthesis

2,2'-Bis(bromomethyl)-1,1'-binaphthalene was prepared according to a literature method [9] from 1-bromo-2-methylnaphthalene. Dichlorobis(benzonitrile)palladium(II) was prepared by a published method [10].

2.1.1. 2,7-Dihydro-1*H*-dinaphtho[*c,e*]tellurepin (**1**)

This compound was prepared by two methods:

Method A. A mixture of freshly crushed and finely ground tellurium powder (1.0 g; 8 mmol) and dry powdered potassium cyanide (0.53 g; 8 mmol) in dry DMSO (30 cm³) was refluxed under nitrogen atmosphere with stirring until almost tellurium was dissolved (~2 h). To

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the resulting solution, diluted with dry DMSO (20 cm³) and cooled to room temperature, was added a solution of 2,2'-bis(bromomethyl)-1,1'-binaphthalene (1.76 g; 4 mmol) in 25 cm³ of dry DMSO dropwise over period of 45 min. The mixture was stirred for 2 h at room temperature and the pale-yellow solution filtered, poured into water (500 cm³), left for 12 h and the extracted with benzene (4 × 50 cm³). The combined extracts was dried (MgSO₄) and filtered. The solvent was removed by a rotary evaporator to give a yellow precipitate. Recrystallization from ethanol gave a yellow solid product in 44% yield (0.27 g), m.p. 132–134 °C.

Method B. Reduction of compound **2** by hydrazine hydrate gave compound **1** in 72% yields and as follow:

Hydrazine hydrate was added to a boiling solution of **2** (0.66 g; 1 mmol) until the orange colour had disappeared. The solution was filtered, then poured into water (100 cm³) and extracted into diethyl ether. The ethereal layer was separated, dried over anhydrous calcium chloride and taken to dryness on a rotary evaporator. The resulting yellow powder, on treatment with charcoal and ether gave yellow solid compound; m.p. 132–134 °C in 72% yield or 0.29 g.

Anal. found: C, 64.56; H, 4.20; Te, 30.65. Calc. for C₂₂H₁₆Te: C, 64.77; H, 3.95; Te, 31.28%. ¹H NMR (DMSO-d₆): 3.74 and 4.07(ABq, 4H, CH₂, J_{AB} 13.0Hz) 6.80–8.20(m, 12H, Ar-H).

¹³C NMR (CDCl₃): 8.9(CH₂), 125.8, 126.1, 126.7, 127.2, 128.1, 128.6, 132.5, 134.1, 136.3, 140.1(aromatic). Mass spectrum: *m/z* (relative intensity): 410 (64%).

2.1.2. 1,1-Diiodo-2,7-dihydro-1H-dinaphtho[*c,e*]telluripin (**2**)

This compound was prepared by two methods:

Method A. A stirred mixture of 2,2'-bis(bromomethyl)-2,2'-dinaphthalene (0.88 g; 2 mmol), tellurium powder (0.255 g; 2 mmol) and sodium iodide (3.0 g; 10 mmol) in 2-methoxyethanol (50 cm³) was heated gently for 4 h. The hot solution was filtered and the filtrate cooled to room temperature during which an orange-red precipitate was formed. Deionized water(500 cm³) was added to cause an additional precipitate. The precipitate was collected and washed several times with water and ethanol, respectively. Recrystallization from 2-methoxyethanol afforded an orange-red solid in 56% yield, m.p. 232 °C.

Method B. Iodine (0.30 g; 1.20 mmol) in dry ether (15 cm³) was added to a well-stirred solution of compound **1** (0.50 g; 1.20 mmol) in dry ether(25 cm³) at room temperature. An orange precipitate was formed immediately. The product was washed with water and ethanol for several times, respectively. Recrystallization from 2-methoxyethanol gave an orange-red solid in 85% yield, m.p. 232–234 °C.

Anal. found: C, 40.16; H, 2.58; I, 38.11, Te, 19.17. Calc. for C₂₂H₁₆TeI₂: C, 39.93; H, 2.44; I, 38.35; Te,

19.28%. ¹H NMR (DMSO-d₆): 4.18 and 4.52(ABq, 4H, CH₂, J_{AB} 13.1 Hz); 6.86–8.21(m, 12H, Ar-H). ¹³C NMR (CDCl₃): 45.3(CH₂), 125.7, 126.2, 126.7, 127.2, 128.1, 128.6, 132.5, 134.2, 136.3, 139.9(aromatic). Mass spectrum: *m/z* (relative intensity): 664 (23%). *A*_M(DMSO): 34.5 ohm⁻¹ cm² mol⁻¹.

2.1.3. 1,1-Dibromo-2,7-dihydro-1H-dinaphtho[*c,e*]telluripin (**3**)

To a solution of compound **1** (0.82 g; 2 mmol) in 20 cm³ of dry ether was added drop by drop a solution of bromine (1.28 g; 8 mmol) in 40 cm³ of dry ether. A fine yellow precipitate was formed immediately and soon settled to give almost colourless solution. Bromine was added in this way until a permanent colour of bromine resulted when an additional drop of bromine was added. The solvent was allowed to evaporate at room temperature and a pale yellow precipitate of compound **3** was obtained. The product was recrystallized from ethanol to give a pale-yellow solid of **3** in 75% yield, m.p. 241–243 °C.

Anal. found: C, 46.36; H, 3.24; Br, 28.48, Te, 22.79. Calc. for C₂₂H₁₆TeBr₂: C, 46.54; H, 2.84; Br, 28.15; Te, 22.41%. ¹H NMR (DMSO-d₆): 4.06 and 4.30(ABq, 4H, CH₂, J_{AB} 13.1 Hz); 6.81–8.18(m, 12H, Ar-H). ¹³C NMR (CDCl₃): 48.8(CH₂), 125.6, 126.0, 126.5, 127.1, 128.2, 128.6, 132.8, 134.3, 136.1, 139.5(aromatic). Mass spectrum: *m/z* (relative intensity): 568 (15%). *A*_M(DMSO): 31.6 ohm⁻¹ cm² mol⁻¹.

2.1.4. 1,1-Dichloro-2,7-dihydro-1H-dinaphtho[*c,e*]telluripin (**4**)

Compound **1** (0.82 g; 2 mmol) was dissolved in dry ether (40 cm³) and dry chlorine gas was bubbled through the solution for 1 h. A pale-yellow precipitate was formed. The solvent was evaporated to dryness and the residue was recrystallized from ethanol to give a white solid in 85% yield, m.p. 280–282 °C.

Anal. found: C, 54.65; H, 3.26; Cl, 14.84, Te, 25.85. Calc. for C₂₂H₁₆TeCl₂: C, 55.81; H, 3.37; Cl, 14.81; Te, 26.65%. ¹H NMR (DMSO-d₆): 4.04 and 4.37(ABq, 4H, CH₂, J_{AB} 13.0Hz); 6.80–8.18(m, 12H, Ar-H). ¹³C NMR (CDCl₃): 52.4(CH₂), 125.9, 126.1, 126.8, 127.3, 128.0, 128.5, 131.9, 134.6, 137.5, 139.8(aromatic). Mass spectrum: *m/z* (relative intensity): 480 (18%). *A*_M(DMSO): 25.4 ohm⁻¹ cm² mol⁻¹.

2.1.5. 1-Methyl-2,7-dihydro-1H-dinaphtho[*c,e*]telluripinium iodide (**5**)

Excess of freshly distilled iodomethane (10 cm³) was placed with compound **1** (0.40 g; 1 mmol) in 30 cm³ of dry ether in a flask flushed with dry nitrogen. An exothermic reaction took place giving a pale-yellow precipitate which on crystallization from ethanol: water(3:1), gave a bright white solid, m.p. 171–173 °C.

Anal. found: C, 49.65; H, 3.16; I, 23.11, Te, 22.75. Calc. for $C_{23}H_{19}TeI$: C, 50.24; H, 3.48; I, 23.08; Te, 23.20%. 1H NMR (DMSO- d_6): 1.30(s, 3H, CH_3); 3.80–4.90 (m, 4H, CH_2); 6.70–8.40(m, 12H, Ar-H). ^{13}C NMR ($CDCl_3$): 6.5(CH_3); 36.8(CH_2), 125.5, 126.2, 126.8, 127.3, 128.2, 128.7, 132.5, 135.8, 137.9, 140.2(aromatic). Mass spectrum: m/z (relative intensity): 552 (7%). A_M (DMSO): $35.5 \text{ ohm}^{-1} \text{ cm}^2 \text{ mol}^{-1}$.

2.1.6. 1-Ethyl-2,7-dihydro-1H-dinaphtho[*c,e*]tellurepinium iodide (6)

This compound was prepared in the way as compound **5** using excess of freshly distilled iodoethane. A white solid was obtained in good yield, m.p. 198 °C.

Anal. found: C, 51.28; H, 3.52; I, 22.03, Te, 21.87. Calc. for $C_{24}H_{21}TeI$: C, 51.12; H, 3.75; I, 22.50; Te, 22.63%. 1H NMR (DMSO- d_6): 1.36(t, CH_3 , 3H); 2.45(q, 2H, CH_2); 4.16–4.80 (m, 4H, CH_2); 6.80–8.20(m, 12H, Ar-H). ^{13}C NMR ($CDCl_3$): 8.5(CH_3); 20.4(CH_2); 35.6(Te- CH_2), 125.6, 126.2, 126.6, 127.1, 128.2, 128.7, 132.4, 134.2, 136.4, 139.4(aromatic). Mass spectrum: m/z (relative intensity): 566 (6%). A_M (DMSO): $33.7 \text{ ohm}^{-1} \text{ cm}^2 \text{ mol}^{-1}$.

2.1.7. Reaction of compound 1 with DDQ and TCQ (7 and 8)

To a warm solution of compound **1** (1.0 g; 2.4 mmol) in acetonitrile (30 cm^3) was added a warm solution of DDQ or TCQ (2.4 mmol) in acetonitrile (20 cm^3). The resulting solution was refluxed under nitrogen atmosphere for 3 h. The volume of the solution was reduced to 10 cm^3 and the residue was left to crystallize. Dark red-brown (**7**) or brown (**8**) solid was obtained.

7: Anal. found: C, 50.56; H, 2.14; Cl, 15.52; N, 5.96; Te, 14.21. Calc. for $C_{38}H_{22}Cl_4N_4O_6Te$: C, 50.69; H, 2.46; Cl, 15.75; N, 6.23; Te, 14.17%.

1H NMR (DMSO- d_6): 3.81 and 4.11 (ABq(broad), 4H, CH_2 , J_{AB} 13.1 Hz); 6.85–8.18(m, 16H, Ar-H). ^{13}C NMR ($CDCl_3$): 33.6(CH_2), 125.5, 126.1, 126.4, 127.5, 128.1, 128.5, 132.5, 136.5, 139.5, 145.3(aromatic); 104.0, 115.6, 127.8, 152.2(hydroquinone). Mass spectrum: m/z (relative intensity): 228 (76%), 410 (56%), 444 (32%).

8: Anal. found: C, 43.62; H, 2.23; Cl, 29.71; Te, 13.35. Calc. for $C_{34}H_{22}Cl_8O_6Te$: C, 43.55; H, 2.36; Cl, 30.24; Te, 13.60%.

1H NMR (DMSO- d_6): 3.86 and 4.14 (ABq(broad), 4H, CH_2 , J_{AB} 13.1 Hz); 6.85–8.21(m, 16H, Ar-H). ^{13}C NMR ($CDCl_3$): 33.2(CH_2), 125.8, 126.2, 126.6, 127.8, 128.2, 128.4, 131.7, 133.8, 136.6, 139.6(aromatic); 117.2, 146.8(hydroquinone). Mass spectrum: m/z (relative intensity): 246 (72%), 410 (45%), 444 (23%).

2.1.8. Bis(2,7-dihydro-1H-dinaphtho[*c,e*]tellurepin)dichloropalladium(II) (9)

Bis(benzonitrile)dichloropalladium(II) (0.384 g; 1 mmol) was dissolved in dry toluene (50 cm^3) and small

amount of insoluble material was filtered off. To the resulting solution was added compound **1** (0.82 g; 2 mmol). The solution was stirred for 24 h at room temperature. Solvent was removed by a rotary evaporator to give an orange precipitate. The precipitate was washed with hexane until no yellow colour appeared in washing. The orange precipitate was dried in vacuo, yield 65%.

Anal. found: C, 53.11; H, 3.02; Cl, 6.75; Te, 24.36. Calc. for $C_{44}H_{32}Cl_2PdTe_2$: C, 53.21; H, 3.24; Cl; 7.14; Te, 25.60%. 1H NMR (DMSO- d_6): 4.24 and 4.48(ABq(broad), 4H, CH_2 , J_{AB} 13.0Hz) 6.81–8.23(m, 12H, Ar-H).

2.2. Physical measurements

1H and ^{13}C NMR spectra were recorded on a Jeol EX-90FT and Bruker MW-250 spectrometers with TMS as an internal reference. Solution conductivities were measured with a Jenway-Pem3 conductivity meter, using a standard conductivity cell with cell constant of 0.68 cm^{-1} . Infrared spectra were recorded with KBr discs in the range of 4000–200 cm^{-1} on a Pye-Unicam SP-300s infrared spectrophotometer. Mass spectra were measured at 70 eV with an MAT 1125 Finnigan mass spectrometer; peaks shown relative to ^{130}Te . Microanalysis for carbon, hydrogen and nitrogen was obtained on a Carlo-Ebra EA1-108 Elemental Analyzer. All melting points were determined by a Gallenkamp melting point apparatus and are uncorrected.

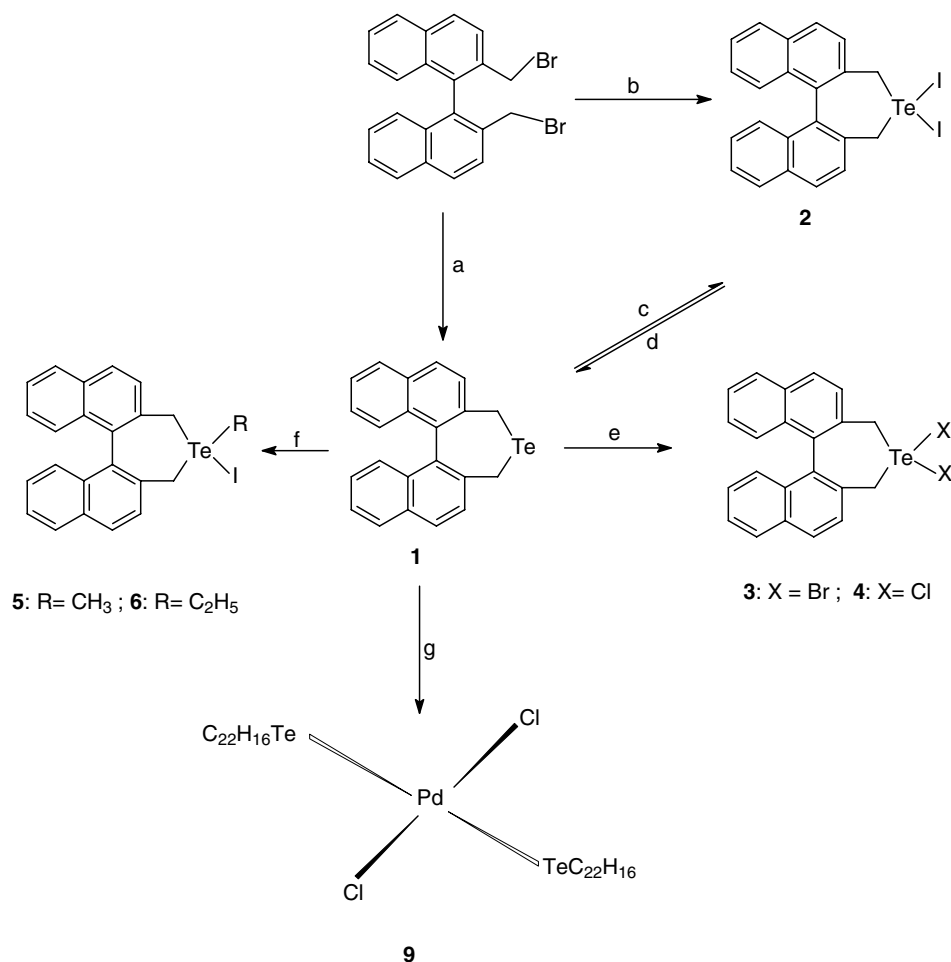
3. Results and discussion

The present work describes the synthesis of new heterocyclic compound containing C-central chiral binaphthyl group (i.e., compound **1**) by reacting 2,2'-bis(bromomethyl)-1,1'-binaphthalene with potassium tellurocyanate [11]. Compound **1** was also prepared by reducing its diiodo(**2**), which in turn can be prepared directly from the treatment of 2,2'-bis(bromomethyl)-1,1'-binaphthalene with tellurium powder and sodium iodide using 2-methoxyethanol as a solvent, Scheme 1.

Compound **1** is quite stable at room temperature and soluble in common organic solvents. This compound, which represents a superior starting material for the preparation of heterocyclic tellurium(IV), was reacted with halogens to afford 1,1-diiodo (**2**), 1,1-dibromo (**3**) and 1,1-dichloro (**4**) derivatives, Scheme 1.

Furthermore, treatment of **1** with iodomethane and iodoethane gave the corresponding tellurepinium iodides **5** and **6**, respectively in good yields, Scheme 1.

The conductivities of compounds **2**, **3**, **4**, **5** and **6** were obtained in DMSO solution. Single concentration data (10^{-3} M) are presented in Section 2. The values of molar conductance approach those expected for 1:1



Scheme 1. Preparative methods for compounds 1–6 and 9. Reagents: (a) KTeCN/DMSO; (b) Te/KI/2-MeOEtOH; (c) I₂; (d) N₂H₄; (e) Br₂ or Cl₂; (f) CH₃I or C₂H₅I; (g) [Pd(PhCN)₂Cl₂].

electrolytes [9,12,13]. Each compound was examined over a range of concentration. In each case a non-linear plot of molar conductance against (concentration)^{1/2} were obtained indicating some degree of ion pairing in DMSO solution. These observations agree well with previous work [4,12].

Infrared spectra of 1–6 displayed common features in certain regions and characteristic bands in the fingerprint and other region. IR spectra show two weak to medium bands due to $\nu_{\text{as}}(\text{C}-\text{Te})$ and $\nu_{\text{s}}(\text{C}-\text{Te})$ generally appeared at the range of 500–530 and 430–480 cm^{-1} , respectively, which agreed well with previous works [4,12]. The dichloro(4) showed a strong band at 320 cm^{-1} , which attributed to $\nu(\text{Te}-\text{Cl})$ [13,14]. The band assigned to $\nu(\text{Te}-\text{Cl})$ was absent in the spectra of compounds 2 and 3.

¹H NMR spectra of compounds 1, 2, 3 and 4 show signals corresponding to methylene and aromatic protons in 1:3 ratio, see Section 2. The methylene protons (2 and 7) give two doublets of a simple AB patterns with ²J coupling constant of ~13.0 Hz. The non-equivalence of the CH₂ protons in 1–4 could be at-

tributed to the very slow or completely hindered inversion of the tellurepin ring which is not planar on account of the bulky tellurium atom, and forced into a somewhat rigid frame by the condensed naphthalene rings. We were unable to study variable-temperature ¹H NMR experiments, for a search of the broadening or even the coalescence of the CH₂AB system, because small amounts of elemental tellurium were always deposited during this experiment. However, the formation of tellurepinium salts 5 and 6, i.e., replacement of one halogen atom by methyl or ethyl group covalently attached to tellurium, showed a remarkable change in the methylene spectrum; it appeared as two AB spectra for the protons at C-2 and C-7 (Section 2). ¹³C NMR data were obtained in CDCl₃ (Section 2). The large variation for carbon atoms bearing tellurium atoms can be attributed to the polarity of the Te–C bond [12,15].

Reaction of compound 1 with DDQ (2,3-dichloro-5,6-dicyano-1,4-benzoquinone) and with TCQ (tetrachloro-1,4-benzoquinone) in acetonitrile afforded compound 7 and 8. The IR spectra of these compounds

show the disappearance of $\nu(\text{CO})$ and the presence of OH group around 3460 and 3400 cm^{-1} , respectively. The broadness of the $\nu(\text{OH})$ IR bands of **7** and **8** may indicate the involvement in hydrogen bonding. Furthermore, a broad band around 600 cm^{-1} was observed in the spectra of **7** and **8**, which is due to Te–O vibration [16,17]. The ^1H NMR spectra of **7** and **8** showed enhancement of aromatic protons with respect to methylene protons (Section 2). This may be attributed to the merge of OH signals with phenyl protons. The methylene protons (2 and 7) give two broad doublets of a simple AB patterns, Section 2.

The ^{13}C NMR spectra of **7** and **8** showed extra signals due to the presence of hydroquinones (see Section 2). Furthermore, no molecular ion was observed in the mass spectra of both complexes. The spectra indicate the presence of ions such as $\text{C}_{22}\text{H}_{16}\text{Te}(\text{OH})_2^+$, $\text{C}_{22}\text{H}_{16}\text{Te}^+$ and the hydroquinone radicals. Reaction of **7** and **8** with FeCl_3 gave a positive test for the presence of phenolic group. It is well known that the addition of quinones to organotellurium compounds, such as TCNE, in some cases prompted formation of telluroxide [18]. Thus, DDQ and TCQ may prompt the formation of telluroxide which in turn react with two moles of the hydroquinone to form 1:2 adduct compounds. Compounds reported as telluroxides were sometimes formulated as $\text{R}_2\text{TeO} \cdot \text{H}_2\text{O}$, and sometimes as $\text{R}_2\text{Te}(\text{OH})_2$ [17,18,19]. Thus, the structure of **7** and **8** need to be interpreted with caution, as it might be doubtful whether hydrated species or hydroxides really exist. Furthermore, the presence of Te–OH or Te–O–Te bonds uncertain. In general, it may be concluded that compound **1** reduces quinones to hydroquinones.

Reaction of **1** with $[\text{PdCl}_2(\text{CNPh})_2]$, which performed in dry toluene in 1:2 molar ratio, yield the orange mononuclear palladium(II) complex (**9**). IR spectrum of complex **9** shows only one $\nu(\text{Pd–Cl})$ at 350 cm^{-1} . This supports a *trans* geometry of complex **9** [20]. Its ^1H NMR spectrum shows broad signals of AB system a δ_{A} 4.24 and δ_{B} 4.48 ppm compared to well resolved AB system at δ_{A} 3.74 and λ_{B} 4.07 ppm for the free ligand, see Section 2.

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