a crystalline precipitate of presumably $B_3H_7{\cdot}N(CH_3)_3$.) The intensity ratio for $B_2H_4{\cdot}2N(CH_3)_3$:$BH_3{\cdot}N(CH_3)_3$ remained 2:1. When the solution was allowed to staad at room temperature for 2.5 h, the signals of $B_4H_8{\cdot}N(CH_3)_3$ had grown in the spectrum and the intensity of the $BH_3{\cdot}N(CH_3)_3$ signal had become greater than that of the $B_2H_4{\cdot}2N(CH_3)_3$ signal. The signal of $B_3H_7{\cdot}N(CH_3)_3$ still remained strong in the spectrum.

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Supplementary Material Available: Two series of spectra for the reaction solutions of $B_3H_7{\cdot}S(CH_3)_2$ + $N(CH_3)_3$ in $S(CH_3)_2$ and of $B_3H_7{\cdot}THF$ + $N(CH_3)_3$ in THF (2 pages). Ordering information is given on any current masthead page.

Contribution from the Faculty of Pharmaceutical Sciences, Nagoya City University, Tanabe-dori, Mizuho-ku, Nagoya 467, Japan, and Department of Chemistry, Gorlaeus Laboratories, Leiden University, 2300 RA Leiden, The Netherlands

Unusual Isomerization Reaction of a Platinum–Dinucleotide Compound

Kenji Inagaki,*[1a] Maarten Alink,[1a] Akinori Nagai,[1a] Yoshinori Kidani,*[1a] and Jan Reedijk[1b]

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It has been generally believed that bifunctional attack of antitumor platinum complex, e.g., *cis*-$Pt(NH_3)_2Cl_2$, on DNA is responsible for an appearance of the biological activities.[2] Much attention has been focused on characterization of platinum–oligonucleotide adducts[3] formed after chelation due to *cis*-$Pt(NH_3)_2Cl_2$. The reaction between a bifunctional platinum complex and nucleic acid base occurs via a two-step mechanism,[4] and the first step is normally a preferential binding to the N7 site of a guanine.[5] The present paper mainly describes results obtained about the second step, i.e., chelation after the first platinum binding. The reaction of $[Pt(R,R\text{-dach})(OH_2)_2]^{2+}$ with r(GpA) (abbreviations: *R,R*-dach, 1*R*,2*R*-cyclohexanediamine; r(GpA), guanylyl(3′-5′)adenosine) results in interbase cross-linked compounds between the guanine and the adenine bases. These chelate compounds are formed via the 1:1 aqua intermediate, Pt(*R,R*-dach)(OH_2)(r(GpA)-*N7(1)*). This process is a normal two-step reaction. An intriguing observation is that one of the chelate compounds, which is likely to be a kinetically preferred species, isomerizes very slowly to give the end products. That is, the isomerization process is a reaction in which the kinetically preferred species is slowly converted to thermodynamically more stable chelates. The aim of the present paper is to introduce such an unusual isomerization reaction. Such an isomerization reaction could not be observed in the case of reaction between r(ApG) and $[Pt(R,R\text{-dach})(OH_2)_2]^{2+}$.

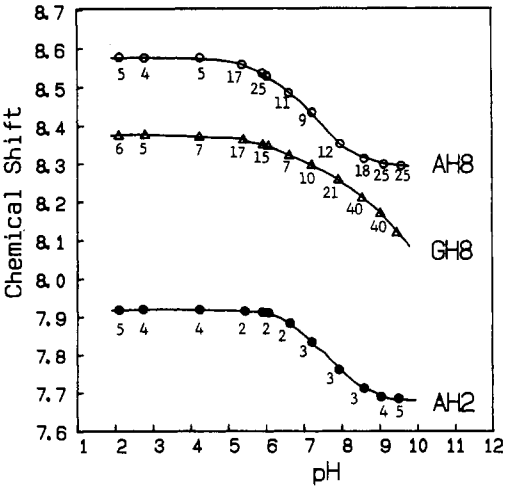

Figure 1. Chemical shift vs pH of the purine base protons of the Pt adduct **4**, Pt(*R,R*-dach)(r(GpA)-*N7(1),N1(2)*). The numbers along the plots indicate the line widths of the signals at half-height (Hz).

The reaction of $[Pt(R,R\text{-dach})(OH_2)_2]^{2+}$ with r(GpA) was followed by means of HPLC as a function of time (10 °C, pH 4.4). The reaction results in four Pt adducts (**1–4**)[6] formed via an aqua 1:1 intermediate. The reaction of Pt(*R,R*-dach)Cl_2 with r(GpA) also results in the same four Pt adducts, in this case without an observable intermediate because the hydrolysis reaction of the dichloro complex is the rate-determining step. The relative ratio of the four Pt adducts was found to be constant throughout the reaction[7] because the isomerization under consideration is very slow at 10 °C. With the reaction at higher temperature, e.g. 37 °C, the relative ratio was almost constant for the first 10 h (see Table I). However, the amount of Pt adduct **4** appeared to decrease thereafter, apparently because of an isomerization reaction. The kinetic parameters obtained under the pseudo-first-order conditions [the concentrations of $[Pt(R,R\text{-dach})(OH_2)_2]^{2+}$ were 10–40 times in excess over those of r(GpA)] are indicated in Table I. The occurring reaction can be described according to the following scheme:

$$\text{r(GpA)} + \text{Pt}(R,R\text{-dach}) \xrightarrow[k_1]{} \text{1:1 aqua intermediate} \xrightarrow[k_c]{} \textbf{1–4} \quad (1)$$

where $k_c = k_{1c} + k_{2c} + k_{3c} + k_{4c}$. Each kinetic constant has been calculated from the relative peak areas of HPLC chromatograms. The kinetic parameters (k_1 and k_c) are in fair agreement with the corresponding parameters reported by Chottard et al.[8] for the reaction between $[cis\text{-}Pt(NH_3)_2(OH_2)_2]^{2+}$ and r(GpA).

In the Pt adducts **2–4**, the binding ratio of $[Pt(R,R\text{-dach})]^{2+}$ to r(GpA) was found to be 1:1, i.e., Pt(*R,R*-dach)(r(GpA)), being calculated from integrations of the H1′ protons of the r(GpA) moiety and the CH protons of the cyclohexane ring in the NMR spectra. Adduct **3** can definitely be assigned to Pt(*R,R*-dach)(r(GpA)-*N7(1),N7(2)*) from evidence of UV, CD (data not

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(7) The relative ratios were calculated from peak areas of the HPLC chromatogram. Although the molar extinction coefficients (at 260 nm) of the Pt adducts and unreacted r(GpA) are not identical, they are expected to be almost the same (Inagaki, K.; Tomita, A.; Kidani, Y. *Bull. Chem. Soc. Jpn.* **1988**, *61*, 2825).
(8) These authors[4c] followed the reaction between r(GpA) and $[cis\text{-}Pt(NH_3)_2(OH_2)_2]^{2+}$ by means of UV spectrometry and HPLC and obtained the values $k_1 = 0.8\ M^{-1}\ s^{-1}$ and $k_c = 4.5 \times 10^{-4}\ s^{-1}$ at 20 °C, pH = 5.2. They isolated *cis*-$Pt(NH_3)_2$(r(GpA)-*N7(1),N7(2)*) (68%) and *cis*-$Pt(NH_3)_2$(r(GpA)-*N7(1),N1(2)*) (32%), and the latter compound corresponds to the adduct **4** in this work.

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Table I. Products Obtained from the Reaction between Pt(*R*,*R*-dach)$(OH_2)_2$ and r(GpA) and Kinetic Constants for the Reaction

abbr	compd	retention time,[a] min	*R* ratio of **1–4**,[b] %	rate const[c] (at 10 °C)
	r(GpA)	19.4		
int	Pt(*R*,*R*-dach)(OH_2)(r(GpA)-*N7(1)*)	15.8		$k_1 = (0.67 \pm 0.07)$ M^{-1} s^{-1}
1	*d*	6.9	3.3–3.6	$k_{1c} = (2.2 \pm 0.3) \times 10^{-5}$ s^{-1}
2	Pt(*R*,*R*-dach)(r(GpA)-*N7(1)*,*N1(2)*)	9.6	6.8–7.5	$k_{2c} = (4.1 \pm 0.5) \times 10^{-5}$ s^{-1}
3	Pt(*R*,*R*-dach)(r(GpA)-*N7(1)*,*N7(2)*)	11.1	36–39	$k_{3c} = (2.2 \pm 0.3) \times 10^{-4}$ s^{-1}
4	Pt(*R*,*R*-dach)(r(GpA)-*N7(1)*,*N1(2)*)	14.0	50–54	$k_{4c} = (3.2 \pm 0.4) \times 10^{-4}$ s^{-1}

[a] The parameters of the HPLC run are as follows: column, Cosmosil $5C_{18}$ (0.46 cm i.d. × 15 cm); detector, UV at 260 nm; flow rate, 1.2 mL/min; mobil phase, linear gradient elution (1%/min) from 0.05 M KH_2PO_4 (pH 4.6) to methanol. [b] Values obtained for the first 10 h of reaction at 37 °C. [c] k_{1c}, k_{2c}, k_{3c}, and k_{4c} are kinetic constants for the formation reactions of **1–4**, respectively, from the aqua intermediate. [d] Compound not definitely determined. From pH–UV and pH–NMR titrations, the pK_a value of the G–N1 site of Pt adduct **1** is found to be 8.2. No protonation at G-N7 and A-N1 is observed.

shown), and NMR spectral data and pK_a values of the G-N1 (pK_a = 8.2) and A-N1 sites (pK_a = 1.5) after platination.[4c] It contains two kinds of rotamers, likely G_{anti}–A_{anti}(75%)[9a] and G_{anti}–A_{syn}-(25%),[9b] because two sets of three protons are observed in the purine base proton region. Adduct **2** can definitely be assigned to Pt(*R*,*R*-dach)(r(GpA)-*N7(1)*,*N1(2)*). This adduct also contains two rotamers,[10a] most likely G_{anti}–A_{anti} (42%)[10b] and G_{anti}–A_{syn} (58%).[10c] From UV, CD, and NMR spectral evidence, it appears that adduct **4** has the same structure as *cis*-Pt$(NH_3)_2$(r(GpA)-*N7(1)*,*N1(2)*), being reported by Chottard et al.[4c] They assigned *cis*-Pt$(NH_3)_2$(r(GpA)-*N7(1)*,*N1(2)*) to G_{syn}–A_{rot} with a rotation of the adenine base about the glycosidic and Pt–A-N1 bonds. Figure 1 shows a pH titration curve of the adduct **4**. No protonation at the G-N7 and A-N1 sites is observed, and deprotonation at G-N1 affects not only the chemical shift of G-H8 but also those of A-H2 an A-H8. This suggests that the deprotonation of G-N1 induces a certain change in conformation of the A base. The G-H8 and A-H8 signals broaden very much at alkaline pH region (see Figure 1).[11] These NMR spectral features are in good agreement with Chottard's data.[8] We therefore assign **4** to an interbase cross-linked compound with G-N7 and A-N1, probably Pt(*R*,*R*-dach)(r(GpA)-*N7(1)*,*N1(2)*) with G_{syn}–A_{rot}. The adduct **1**, unfortunately, could not be sufficiently characterized because of the very small amount.

As described above, the relative ratio of the four platinum adducts was almost constant for the first 10 h (at 37 °C). But the subsequent incubation results in a decrease of **4** and an increase of **1–3** even after disappearance of free r(GpA) and the aqua 1:1 intermediate. This strongly points toward an isomerization from **4** to **1–3**. Figure 2 shows a time dependence of the conversion reaction from **4** to **1–3**. The half-life of **4** was 8.8 h at 60 °C. The relative ratio of the end products **1–3** was constant throughout the isomerization reaction and also similar to the values obtained from the reaction of $[Pt(R,R\text{-dach})(OH_2)_2]^{2+}$ with r(GpA). The adduct **4** decreases according to the first-order reaction. Although the adducts **1–3** are formed without an observable intermediate, they are likely to be formed via the aqua intermediate, Pt(*R*,*R*-dach)(OH_2)(r(GpA)-*N7(1)*), i.e., according to the following scheme:

$$\mathbf{4} \underset{k_{-s}}{\overset{k_s}{\rightleftharpoons}} \text{aqua intermediate} \xrightarrow{k_{sc}} \mathbf{1\text{–}3} \qquad (2)$$

The reaction scheme corresponds to the formation reaction of the four Pt adducts from the aqua intermediate in eq 1. If solvolysis reaction of **4** is very slow, i.e. $k_{-s} \gg k_s$ or $k_{-s} + k_{sc} \gg k_s$, the

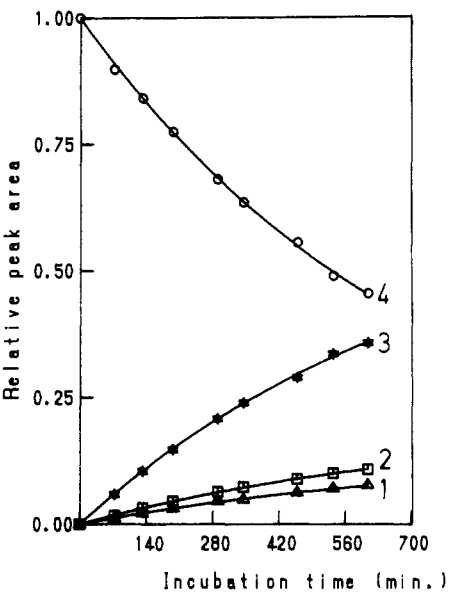

Figure 2. Time dependence of the isomerization reaction of the Pt adduct **4** at 60 °C in water. [**4**] = 3.0 × 10^{-4}; pH = 5.5.

apparent rate constant, $k_{obs(s)}$, in reaction scheme 2 is given by $k_{obs(s)} = k_s k_{sc}/(k_{-s} + k_{sc})$, where $k_{sc} = k_{1c} + k_{2c} + k_{3c}$ and $k_{-s} = k_{4c}$. The following values were calculated from the data in Table I and from the values[12] of $k_{obs(s)}$ at 37, 61, and 77 °C: $k_{obs(s)} = 9.7 \times 10^{-8}$ s^{-1}, $k_s = 2.0 \times 10^{-7}$ s^{-1}, and $k_{sc} = 2.8 \times 10^{-4}$ s^{-1} at 10 °C. The kinetic parameters (supplementary material Table SI) already indicate that the aqua intermediate is difficult to be detected because $k_{-s} + k_{sc} \gg k_s$. Moreover, thermodynamic parameters calculated from an Eyring plot suggest that the isomerization reaction proceeds via an association mechanism ($\Delta H^{\ddagger} = 78.5$ kJ mol^{-1} and $\Delta S^{\ddagger} = -103$ J K^{-1} mol^{-1}).[13] The aquation path in the reaction appears to be supported by the decrease in rate when the solvent is substituted for a water–ethanol mixture (the values of $k_{obs(s)}$ (×10^5 s^{-1}) at 60 °C are 2.2, 1.4, and 1.0 at 0%, 25%, and 50% ethanol, respectively). The rate of the isomerization reaction tends to be slightly speeded up with lowering the pH (the values of $k_{obs(s)}$ at 37 °C (×10^6 s^{-1}) are 3.4, 2.7, 2.2, 1.7, and 0.7 at different pHs 1.8, 2.5, 4.4, 6.2, and 9.1, respectively). The relative ratio of the final Pt adducts **1–3**—being obtained from the isomerization reaction—also changes as a function of pH. The formation of **1** and **2** tends to be suppressed with lowering the pH. Especially the formation of **2** is strongly

(9) (a) NMR (ppm): A-H8, 9.34; G-H8, 8.55; A-H2, 8.34. The *T*1 relaxation times of A-H8 and G-H8 are 1.05 and 0.75 s^{-1} at 26 °C and 400 MHz, respectively. (b) NMR (ppm): A-H8, 9.14; G-H8, 8.54; A-H2, 8.30. The *T*1 relaxation times of A-H8 and G-H8 are 1.30 and 0.54 s^{-1} at 26 °C, respectively.

(10) (a) Chemical shift data for **2** are in good agreement with those of *cis*-Pt$(NH_3)_2$(d(GpA)-*N7(1)*,*N1(2)*) with G_{anti}–A_{syn} (30%) and G_{anti}–A_{anti} (70%): Dijt, F. J.; Chottard, J. C.; Girault, J. P.; Reedijk, J. *Eur. J. Biochem.* **1989**, *179*, 333. The data for *cis*-Pt$(NH_3)_2$(d-(GpA)-*N7(1)*,*N1(2)*) are indicated within parentheses. (b) NMR (ppm): A-H8, 8.34 (8.40); G-H8, 7.92 (7.91); A-H2, 8.43 (8.48). (c) NMR (ppm): A-H8, 8.26 (8.25); G-H8, 8.64 (8.61); A-H2, 8.68 (8.61).

(11) The G-H1′ signal also broaden from 4 (pH 2–6) to 33 Hz (pH 9.3).

(12) The $K_{obs(s)}$ value at 10 °C was estimated from extrapolation of $k_{obs(s)}$ vs temperature because the conversion reaction of 4 to **1–3** was too slow to measure at low temperature and an Eyring plot using apparent kinetic constant ($k_{obs(s)}$ showed a clear straight line (the values of $k_{obs(s)}$ are 1.5 × 10^{-6}, 1.5 × 10^{-5}, and 5.7 × 10^{-5} s^{-1} at 37, 61, and 77 °C (pH 6; solvent 9% methanol), respectively).

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suppressed at low pH (supplementary material Figure S1). This is likely to be due to the fact that protonation at A-N1 of the aqua intermediate inhibits the formation of **2**. The competitions at A-N1 between platinum and proton are also expected to occur in the reaction forming **4** from the aqua intermediate, i.e. k_{-5}, and this is likely to lead a promotion of $k_{obs(s)}$ observed at low pH.

The isomerization from **4** to **1-3** is enhanced in a presence of halogen ions. In the HPLC chromatogram, an intermediate peak, presumably Pt(*R*,*R*-dach)(X)(r(GpA)-*N*7(*1*)), X = Cl, Br, and I, has been observed. The observed rate constant for the conversion of **4** shows a linear dependence upon an excess of halogen ion concentration ([X] = 0.01-0.2 M) and a nonzero intercept at [X] = 0. The data fit well in a two-term rate expression ($k_{obs} = k_{obs(s)} + k_X[X]$), which is the well-known rate law for the substitution reactions of the square-planar platinum complex. The rate constants obtained from the intercepts were found to agree within experimental error with the rate constant obtained in an absence of halogen ions. The result also supports the solvolytic path in the isomerization reaction. The values[14] of k_X increases in the order $Cl^- < Br^- < I^-$.

In conclusion, the chelate formation reaction between [Pt(*R*,*R*-dach)]$^{2+}$ and r(GpA) proceeds according to the following reaction scheme (in the absence of halogen ions).

$$\text{r(GpA)} + \text{Pt}(R,R\text{-dach})(\text{OH}_2)_2 \xrightarrow{k_1} \text{aqua intermediate} \xrightarrow{k_c} \textbf{1-4}$$

$$\textbf{4} \underset{k_{-4}\ (\text{fast})}{\overset{k_4\ (\text{slow})}{\rightleftharpoons}} \text{aqua intermediate} \xrightarrow[\text{fast}]{k_{ac}} \textbf{1-3}$$

The reverse reaction (from **1-3** to **4**) was not observed. It is worth noting that the same reaction scheme has also been observed in the reaction between *cis*-[Pt(NH$_3$)$_2$(OH$_2$)$_2$]$^{2+}$ and r(GpA) and d(GpA) and that the conversion reaction such as **4** → **1-3** could not be observed in the case of reaction between [Pt(*R*,*R*-dach)(OH$_2$)$_2$]$^{2+}$ and r(ApG) (unpublished observation).

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Supplementary Material Available: Table SI, giving kinetic data for the isomerization reaction, and Figure S1, showing the relative ratio of the final Pt adducts, **1-3**, as a function of pH (2 pages). Ordering information is given on any current masthead page.

(14) $k_{Cl} = 6.7 \times 10^{-5}$ M^{-1} s^{-1}, $k_{Br} = 4.0 \times 10^{-4}$ M^{-1} s^{-1}, and $k_I = 8.1 \times 10^{-3}$ M^{-1} s^{-1} at 37 °C.

Contribution from the Department of Chemistry,
State University of New York at Buffalo,
Buffalo, New York 14214

Preparation and Characterization of *trans*-Bis(α-dioximato)ruthenium Complexes

James G. Muller and Kenneth J. Takeuchi*

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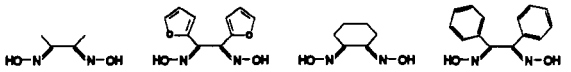
The synthesis and characterization of transition-metal complexes that contain a ruthenium metal center coordinated to chelating nitrogen donor ligands have been subjects of considerable interest in our laboratory[1-6] and elsewhere.[7-19] In addition, the coordination chemistry of chelating α-dioxime ligands has been extensively investigated with first-row transition metals,[22-26] where *trans*-bis(α-dioximato) transition-metal complexes have been utilized as analytical reagents,[20,21,29] models for biological systems such as vitamin B$_{12}$,[30-32] dioxygen carriers,[33,34] and catalysts in chemical processes.[35-38] The only examples of *trans*-bis(α-di-

$$\text{Ru(NO)Cl}_3\cdot 5\text{H}_2\text{O} + 2.1\text{LH}_2 \xrightarrow[\text{EtOH}]{\Delta} \text{Ru(LH)}_2\text{(NO)Cl}$$



LH_2 = DMGH_2 , DFGH_2 , NOXH_2 , DPGH_2

Figure 1. Reaction scheme for the synthesis of *trans*-bis(α-dioximato)-ruthenium complexes. $DMGH_2$ = dimethylglyoxime, $DFGH_2$ = α-furil dioxime, $NOXH_2$ = 1,2-cyclohexanedione dioxime, and $DPGH_2$ = diphenylglyoxime.

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