1. Complete discussion of assignments determined from 1D and 2D NMR data.

HH1 Conformer of $Me_2ppzPt(GpG)$. An NOE cross-peak between the two H8 signals of this conformer was observed; this effect is characteristically found for HH forms.[1] The more upfield H8 signal (8.62 ppm) also showed NOE cross-peaks to two signals; these signals (at 4.22 and 4.81 ppm) were connected by NOE and COSY cross-peaks. An NOE cross-peak between the signal at 4.22 ppm and a signal at 5.93 ppm was also found. The signal at 4.81 ppm showed NOE and COSY cross-peaks to a resonance at 4.25 ppm which had NOE and COSY cross-peaks to signals at 3.81 and 3.98 ppm. These two signals (at 3.81 and 3.98 ppm) were connected in the NOESY and COSY spectra. On the basis of these observations and the chemical shifts of these resonances,[2] the signals at 5.93, 4.22, 4.81, and 4.25 ppm were assigned to H1', H2', H3', and H4', respectively. (The resonances at 3.81 and 3.98 ppm were assigned as the H5'/H5" signals.) Observation of an H8-H2' NOE cross-peak, and the absence of any such cross-peak between the H8 and H1' signals, is characteristic of an *anti* conformation.[3,4] G residues of *cis*-PtA_2(dinucleotide) HH forms typically retain the *anti* conformation found in B-DNA.[1,5-7] The assigned H1' signal (at 5.93 ppm) is a singlet, indicating that the sugar has an N-pucker.[8] This more upfield H8 signal, and its respective sugar signals, are assigned to the 5'-G since the sugar moiety of the 5'-G residue adopts the N-pucker for all known N7-Pt-N7 intrastrand cross-linked adducts.[1,6,9,10] Coupling between the assigned H3' resonance of this residue and the ^{31}P NMR signal for this form was found in an ^{1}H-^{31}P HMBC spectrum and thereby confirms the 5'-G residue assignment. In this type of experiment with nucleic acids, H3'-^{31}P coupling is observed for the 5'-residue while H4'/H5'/H5"-^{31}P coupling is found for the 3'-residue.[2,11,12]

The more downfield H8 signal (8.78 ppm), which must be the 3'-G H8 signal, showed an NOE cross-peak to a sugar signal at 4.42 ppm. This ribose signal was assigned to the 3'-G H2' signal since it was connected by NOE and COSY cross-peaks to an H1' signal at 5.83 ppm. The observed H8-H2' NOE cross-peak and the absence of an H8-H1' NOE cross-peak are consistent with an *anti* G, which, as mentioned above, is typically observed for HH forms of *cis*-PtA_2(dinucleotide) complexes. The H1' signal (at 5.83 ppm) was a doublet; this splitting is characteristic of an S-sugar pucker.[8] The signal at 4.42 ppm was also found to have cross-peaks to a resonance at 4.33 ppm in both the NOESY and COSY spectra. NOE and COSY cross-peaks were observed between the 4.33 ppm and 4.23 ppm signals. The peak at 4.23 ppm also showed NOE and COSY cross-peaks with signals at 3.97 and 4.06 ppm, which were connected to one another in the NOESY and COSY spectra. From these observations, the 3'-G signals at 4.42, 4.33, and 4.23 ppm were assigned to H2', H3', and H4', respectively. The resonances at 3.97 and 4.06 ppm were assigned to the H5'/H5" signals. These two signals showed coupling with the ^{31}P signal of this form in the ^{1}H-^{31}P HMBC experiment; H5'-/H5"-^{31}P coupling is expected for the 3'-residue.

ΔHT1 Conformer of $Me_2ppzPt(GpG)$. No H8-H8 NOE cross-peak was observed between the H8 signals (7.86, 7.91 ppm) of this conformer; the absence of such a cross-peak is consistent with an HT arrangement of the G bases.[13,14] The more upfield H8 peak had an NOE cross-peak to a sugar signal at 3.54 ppm, which showed NOE and COSY cross-peaks with resonances at 4.53 and 4.14 ppm. Both these signals (at 4.53 and 4.14 ppm) showed NOE cross-peaks to a peak at 5.82 ppm. A 5.82-4.53 ppm COSY cross-peak was also found. Thus, the peaks at 5.82, 4.53, 3.54, and 4.14 ppm were assigned to the 5'-G H1', H2', H3', and H4' signals, respectively. The coupling observed between the H3' resonance and the ^{31}P NMR signal of this form in the ^{1}H-^{31}P HMBC spectrum is consistent with a 5'-residue. The observed H8-H3' NOE cross-peak and the absence of H1' coupling for the H1' resonance (found to be a singlet in the 1D spectrum) are consistent with an N-sugar pucker.[2,8] The absence of an H8-H1' NOE cross-peak suggests that this 5'-G is *anti*.[2-4]

The more downfield H8 signal, which must be from the 3'-G, showed an NOE cross-peak

to a doublet at 5.64 ppm, which had NOE cross-peaks to signals at 5.15 and 4.04 ppm. A 5.64-5.15 ppm COSY cross-peak was also found. The signal at 5.15 ppm had a COSY cross-peak to a resonance at 4.94 ppm which, in turn, showed NOE and COSY cross-peaks with the resonance at 4.04 ppm. This peak (at 4.04 ppm) was connected to H5'/H5" signals at 3.72 and 3.87 ppm. Therefore, the signals at 5.64, 5.15, 4.94, and 4.04 ppm were assigned to H1', H2', H3', and H4', respectively. The observation of an intraresidue H8-H1' NOE cross-peak suggests that this G is *syn*.[2] The H1' doublet coupling pattern is characteristic of an S-sugar pucker,[8] typically observed for the 3'-G of GpG adducts.[14] However the small $^3J_{H1'-H2'}$ (2.2 Hz) observed for this H1' signal suggests that this sugar residue is not strictly S, but possesses some N character as well.[5,10] The coupling of assigned H4' and H5'/H5" peaks with the ^{31}P NMR resonance of this form, observed in the ^1H-^{31}P HMBC experiment, confirm the 3'-G residue assignment.

HH1 Conformer of Me$_2$ppzPt(d(GpG)). An NOE cross-peak was observed between the two H8 signals of the HH1 form. The more upfield H8 signal (8.51 ppm) showed a strong NOE cross-peak to a signal at 2.42 ppm and a weaker cross-peak to a resonance at 2.73 ppm. NOE and COSY cross-peaks were observed between the signals at 2.42 and 2.73 ppm, the latter showing a cross-peak to a doublet at 6.16 ppm in both the NOESY and COSY spectra. A 6.16-4.05 ppm cross-peak was also found in the NOESY spectrum. The H8 signal at 8.51 ppm also showed a strong NOE cross-peak to a signal at 5.01 ppm. This signal, in turn, had COSY cross-peaks to the signals at 2.73, 2.42, and 4.05 ppm. From these cross-peaks and the shifts of these signals, the resonances at 6.16, 2.42, 2.73, 5.01, and 4.05 ppm were assigned to the H1', H2', H2", H3', and H4' signals, respectively. The NOE cross-peaks between the H8 and H2'/H2" signals and the absence of an H8-H1' cross-peak are consistent with an *anti* G.[2-4] The doublet coupling of the H1' signal and the intraresidue H8-H3' NOE cross-peak suggest an N-sugar pucker,[2,8] consistent with a 5'-G.[1,6,9,10] This residue assignment is confirmed by the coupling observed between the assigned H3' signal (5.01 ppm) and the ^{31}P NMR signal of this form in the ^1H-^{31}P HMBC spectrum. (The distinction between H2' and H2" signals was based on relative intensities of observed NOE cross-peaks to the H8 signal; typically, H8-H2' NOE cross-peaks are stronger than H8-H2". Moreover, H1'-H2" NOE cross-peaks are stronger than H1'-H2' cross-peaks, and H1'-H2' COSY cross-peaks are generally not observed for an N-sugar.[2])

The more downfield of the two H8 signals (8.93 ppm) must be the 3'-G H8. It showed NOE cross-peaks to signals at 2.40 and 2.50 ppm, the latter cross-peak being the stronger of the two. NOE and COSY cross-peaks connected the 2.40 and 2.50 ppm signals which, in turn, had NOE and COSY cross-peaks to a quartet at 6.21 ppm; the 2.40-6.21 ppm NOE cross-peak was strong, while the 2.50-6.21 ppm NOESY cross-peak was comparatively weak. Furthermore, both signals (at 2.40 and 2.50 ppm) showed NOE cross-peaks to the signal at 4.63 ppm. A 2.50-4.63 ppm COSY cross-peak was also observed. The signal at 4.63 ppm had NOE and COSY cross-peaks with a signal at 4.15 ppm. Thus, from these observed cross-peaks and chemical shifts, the signals at 6.21, 2.50, 2.40, 4.63, and 4.15 ppm were assigned to the 3'-G H1', H2', H2", H3', and H4' signals, respectively. The intranucleotide H8-H2'/H2" NOE cross-peaks and lack of intranucleotide H8-H1' NOE cross-peak suggest an *anti* G.[2-4] The quartet coupling of the 3'-G H1' signal (6.21 ppm) is typical of an S-sugar.[8]

The NOE effects presented in the above discussion are based on NOESY data. NOE cross-peaks found in the ROESY experiment agree with the conformational assignments made for this conformer. For example, an H8-H8 NOE was also found in the ROESY spectrum and is thereby consistent with the HH arrangement of the bases in this form. Both these H8 signals exhibited stronger NOE cross-peaks with their respective H2' resonances while the analogous effects with the H1' signals were weak; these observations suggest that both residues are *anti*.[2-4] The observation of weak H8-H1' NOE cross-peaks in the ROESY spectrum also confirmed H1' signal assignments deduced from the NOESY and COSY data. Lastly, the 5'-G H8 signal showed a strong NOE cross-peak to its respective H3' peak, consistent with an N-sugar pucker conformation for the 5'-residue.

HH2 Conformer of Me_2ppzPt(d(GpG)). Because the H8 signals for this conformer are small and are not well dispersed at the 5 °C collection temperature, it is difficult to determine whether the apparent "cross-peak" in the NOESY spectrum is a true cross-peak or simply the result of noise. However, an NOE was observed between these two H8 signals in 1D NOE experiments conducted at 5 °C. In 2D spectra, the more upfield H8 signal (8.71 ppm) showed NOE cross-peaks with three ribose signals (at 3.01, 2.77, and 4.83 ppm). The 3.01 and 2.77 ppm signals had NOE cross-peaks to a resonance at 6.15 ppm; this signal (6.15 ppm) showed an NOE cross-peak to a resonance at 4.05 ppm. The signal at 4.83 ppm showed a COSY cross-peak to the signal at 2.77 ppm, and NOESY cross-peaks to the signals at 3.07 and 2.77 ppm. From these observations, the signals at 6.15, 3.01, 2.77, 4.83, and 4.05 ppm were assigned to H1', H2', H2", H3', and H4', respectively. These signals are all assigned to the 5'-G since the observed H8-H3' NOE cross-peak and the coupling of the H1' signal indicate an N-sugar pucker.[1,2,6,8-10] The observed coupling between the H3' signal of this residue and the ^{31}P NMR signal of this form in the ^{1}H-^{31}P HMBC experiment confirms the 5'-G residue assignment. This residue is *anti* since an H8-H2' NOE cross-peak is observed, while an H8-H1' NOE cross-peak is not seen.[1-4]

No H8-sugar cross-peaks were observed for the 3'-G H8 signal (8.78 ppm) of the HH2 conformer in the NOESY spectrum. Monitoring the reaction with time allowed assignment of a quartet at 6.11 ppm as the 3'-G H1' signal. (A very weak cross-peak between the signals at 8.78 and 6.11 ppm in the ROESY spectrum confirms the H1' signal assignment). The coupling pattern of this H1' signal is characteristic of an S-sugar pucker.[8] This H1' signal showed NOE cross-peaks to signals at 4.38 and 2.61 ppm, and COSY cross-peaks to the latter signal (2.61 ppm) and a resonance at 2.12 ppm. Cross-peaks between the signals at 2.61 and 2.12 ppm were found in the NOESY and COSY spectra. The 2.12 ppm signal showed NOE and COSY cross-peaks with a signal at 4.60 ppm, which had NOE and COSY cross-peaks to the signal at 4.38 ppm. Signals at 2.12, 2.61, 4.60, and 4.38 ppm were assigned as the 3'-G H2', H2", H3', and H4' signals, respectively. Like the NOESY data, an H8-H8 NOE cross-peak could not be clearly made out in the ROESY spectrum, and very few NOE cross-peaks were found between the H8-sugar signals. However, the same conclusions, as those based on the NOESY data, concerning the conformational features of this form are drawn from the ROESY spectrum (e.g., the more upfield H8 signal showed strong intraresidue NOE cross-peaks to the H2' and H3' peak, and a very weak NOE to the H1' signal; these observations suggest the 5'-G is *anti* and its sugar moiety adopts the N-pucker conformation.[2-4])

ΔHT1 Conformer of Me_2ppzPt(d(GpG)). No H8-H8 NOE cross-peak was observed for the ΔHT1 conformer. The more upfield H8 signal (7.78 ppm) showed a NOESY cross-peak to a signal at 2.83 ppm. NOE and COSY cross-peaks were observed between the signal at 2.83 ppm and a signal at 2.47 ppm. The latter signal had NOE and COSY cross-peaks to a resonance at 6.08 ppm which, in turn, showed an NOE cross-peak to a signal at 4.00 ppm. These signals at 6.08, 2.83, 2.47, and 4.00 ppm were therefore assigned to H1', H2', H2", and H4', respectively. The absence of an H8-H1' NOE cross-peak, but the presence of an H8-H2' NOE cross-peak, suggests an *anti* residue,[1-4] while the doublet coupling of the H1' signal is consistent with an N-sugar pucker;[8] these signals are assigned to the 5'-G.[1,2,6,9,10] The H3' signal of this residue could not be assigned most probably because it was under the HOD peak. Since ^{1}H-^{31}P coupling, characteristic of a 3'-residue, was found for the other G of this form (below), the 5'-G residue assignment is confirmed.

The more downfield H8 signal (7.90 ppm), belonging to the 3'-G, showed a strong NOE cross-peak to a signal at 6.00 ppm. This signal at 6.00 ppm showed NOE and COSY cross-peaks to two signals (at 2.41 and 3.25 ppm); the 6.00-2.41 ppm NOE cross-peak was stronger than the 6.00-3.25 ppm NOE cross-peak. An NOE cross-peak between the signal at 6.00 ppm and a peak at 3.97 ppm was also found. The signal at 3.97 ppm showed NOE cross-peaks with a resonance at 4.92 ppm which, in turn, had NOE cross-peaks with the signals at 2.41 and 3.25 ppm. From these observations, these signals (at 6.00, 3.25, 2.41, 4.92, and 3.97 ppm) were assigned to H1',

H2', H2", H3', and H4', respectively. The H8-H1' NOE cross-peak suggests that this residue is *syn*.[2] The doublet of doublets coupling observed for the H1' resonance is characteristic of an S-sugar pucker;[8] however this signal exhibits a relatively small $^3J_{H1'-H2'}$ (3.1 Hz), and therefore suggests that this sugar moiety may also possess some N character.[5,10] The NOE cross-peaks found in the ROESY spectrum likewise agree with the assignment of the ΔHT1 form. For example, the HT arrangement of the bases of this form is supported by the absence of an observable NOE cross-peak between these two relatively upfield-shifted H8 signals. Additionally, the H8-sugar NOE cross-peaks found in the ROESY spectrum support the *syn* and *anti* assignment for the 3'-G and 5'-G, respectively. (A strong H8-H1' NOE cross-peak was found while only a weak H8-H2' NOE was observed for the 3'-G.[2] In comparison, the 5'-G H8 signal showed a strong NOE cross-peak to its respective H2' resonance and a weaker effect to its respective H1' peak.[2-4] An additional H8-sugar NOE cross-peak (7.78-2.47 ppm), not observed in the NOESY spectrum, was found in the ROESY data; this cross-peak confirms the assignment of the signal at 2.47 ppm to the 5'-G H2" signal. The ROESY spectrum also showed cross-peaks between a signal at 3.63 ppm and the assigned H1' and H4' signals of this residue. A 3.63-3.53 ppm NOE cross-peak was also found. These signals, at 3.63 and 3.53 ppm, are most likely the H5'/H5" signals. These resonances also showed coupling to the ^{31}P NMR signal of this form in the ^{1}H-^{31}P HMBC experiment; such coupling is expected for the 3'-G and thus agrees with the residue assignment.)

2. Further discussion of HH1 and HH2 assignment for Me$_2$ppzPt(d(GpG)).

Some of the biggest shift differences between conformers involve the H8 signals. However, direct comparison of H8 shifts between *cis*-PtA$_2$(dinucleotide) conformers is not straightforward because these shifts are strongly influenced by base canting;[1,7,13,14] the identity of both the carrier ligand and the dinucleotide (e.g., d(GpG) vs. GpG) can affect the direction and degree of base canting. The H8 shift is affected by the ring-current effects of the cis G base[7] and also by the magnetic anisotropy of the platinum atom.[15-18] Thus, we compared shifts of sugar ^{1}H and ^{31}P and NMR signals of these species with those of the established **Bip**Pt(d(GpG)) HH conformers.[1,13]

Shifts of sugar ^{1}H NMR signals were examined since the sugar protons are well removed from the base canting effects. Similar trends were noted between the 5'-G H2', 5'-G H2" and 3'-G H4' signals for the HH1 and HH2 forms of the **Me$_2$ppz**Pt(d(GpG)) and **Bip**Pt(d(GpG)) complexes. While the 5'-G H2' signal was more downfield than the 5'-G H2" signal for the HH2 conformer of (*R,S,S,R*)-**Bip**Pt(d(GpG)),[1] the reverse was found for the (*R,S,S,R*)-**Bip**Pt(d(GpG)) and (*S,R,R,S*)-**Bip**Pt(d(GpG)) HH1 forms.[1,13] This pattern was also found for the two **Me$_2$ppz**Pt(d(GpG)) HH conformers (i.e., the 5'-G H2' signal of the minor HH form was more downfield than its 5'-G H2" signal, while the 5'-G H2" was more downfield than the 5'-G H2' for the major HH form). A rather downfield-shifted H4' signal (4.46 ppm) was reported for the 3'-G of the **Bip**Pt(d(GpG)) HH2 conformer;[1] a similarly distinctive downfield-shifted 3'-G H4' signal (4.38 ppm) is observed for the minor **Me$_2$ppz**Pt(d(GpG)) HH form (more typical 3'-G H4' shifts are closer to ~4.1-4.2 ppm in d(GpG) cross-linked adducts[1,13,14]). This comparison of the sugar ^{1}H NMR signal shifts also indicates that the major HH conformer is HH1, while the minor HH form is HH2.

The ^{31}P NMR signal of the (*R,S,S,R*)-**Bip**Pt(d(GpG)) HH forms are downfield of the free d(GpG) resonance, and the signal of the HH2 conformer is more downfield than that of the HH1 conformer.[1,13] This same shift relationship was found for (*R,R*)-**Me$_4$DAB**Pt(d(GpG)).[11] If this pattern (i.e., the ^{31}P NMR signal of the HH2 form downfield to that of the HH1 form) holds true for the **Me$_2$ppz**Pt(d(GpG)) complex, then the more dominant **Me$_2$ppz**Pt(d(GpG)) HH form is HH1, and the minor HH form is HH2. These ^{31}P NMR shift comparisons support the conformer assignment based on NOE data.

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Table S1. Conformer Distribution As a Function of pH

complex	pH	HH1 %	HH2 %	ΔHT1 %
Me$_2$ppz Pt(d(GpG))				
	4.0	50	20	30
	7.3	50	20	30
	10.3	50	20	30
	10.1	38	<1	62
	(at high pH for 9 d)			
	3.0	38	<1	62
	3.3	40	15	45
	(at low pH for 2 d)			
	3.4	45	17	38
	(at low pH for 11 d)			
Me$_2$ppz Pt(GpG)				
	3.7	86		14
	6.9	86		14
	10.0	86		14
	9.8	63		37
	(at high pH for 8 d)			
	3.5	63		37
	3.7	75		25
	(at low pH for 2 d)			
	3.8	70		30
	(at low pH for 11 d)			

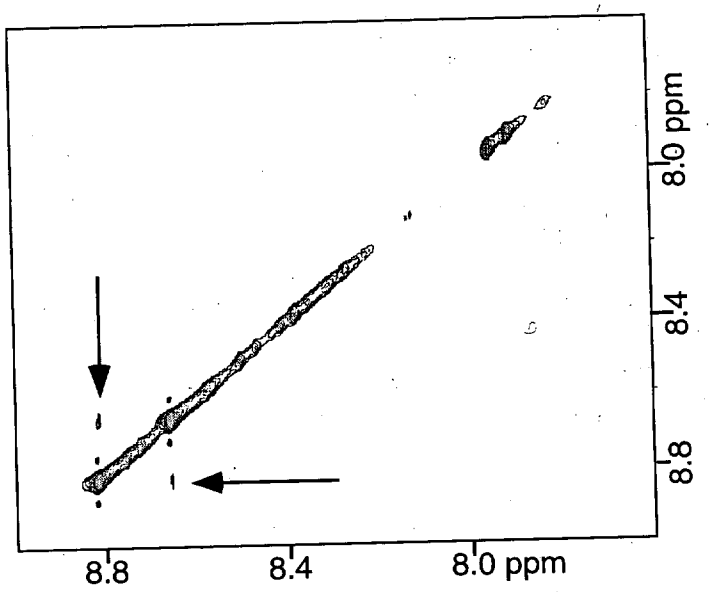

Figure S1. H8 region of **Me_2ppz**Pt(GpG) NOESY spectrum, pH 3.7, 5 °C, D_2O. Arrows mark NOE cross-peak observed for the HH1 conformer.

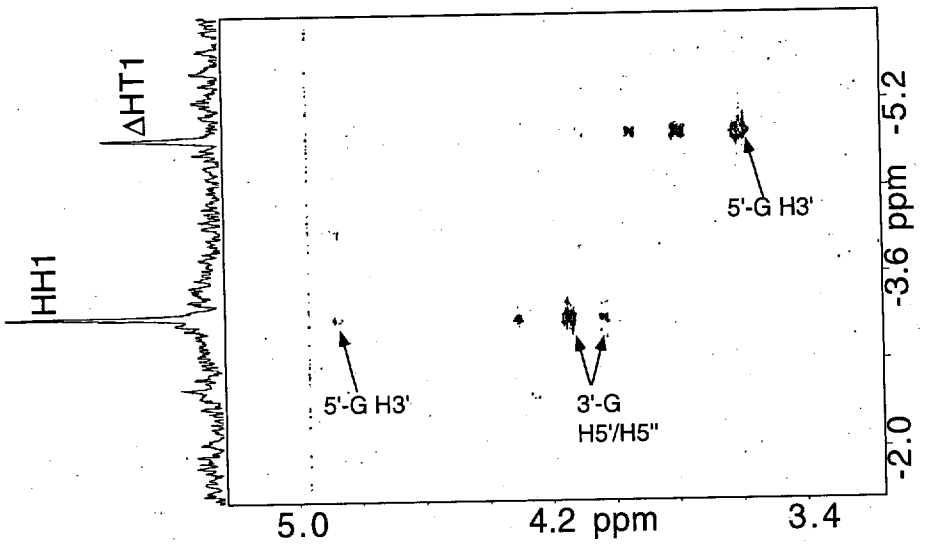

Figure S2. ^{1}H-^{31}P HMBC spectrum of **Me$_2$ppz**Pt(GpG) collected at 5 °C, pH ~4.

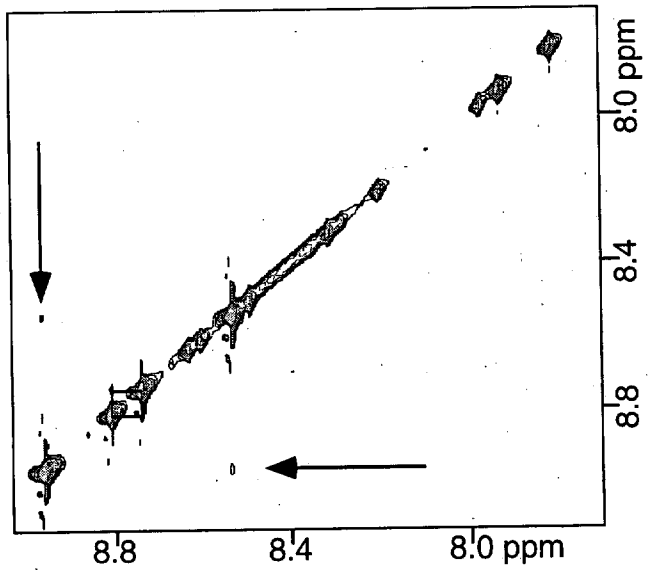

Figure S3. H8 region of **Me$_2$ppz**Pt(d(GpG)) NOESY spectrum, pH 6.5, 5 °C, D_2O. Arrows point to NOE cross-peak for the HH1 conformer and box marks NOE cross-peak for HH2 which was later confirmed in 1D NOE experiments.

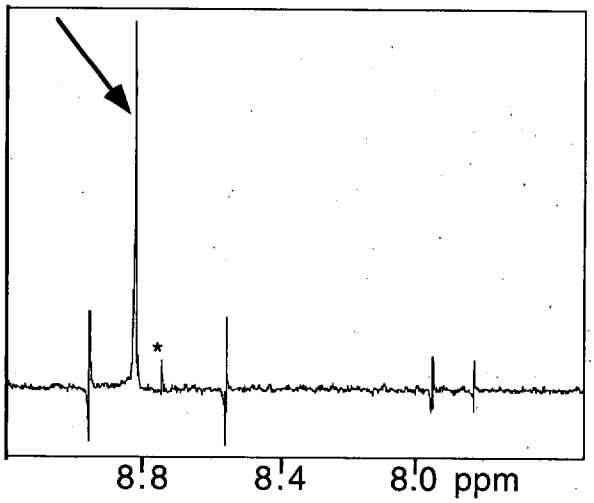

Figure S4. Difference NMR spectrum, 1D NOE experiment with **Me$_2$ppz**Pt(d(GpG)), pH 3.3, 5° C, D_2O. Arrow indicates irradiated peak, asterisk marks signal which experiences NOE. Peaks with positive and negative components arise from substraction error.

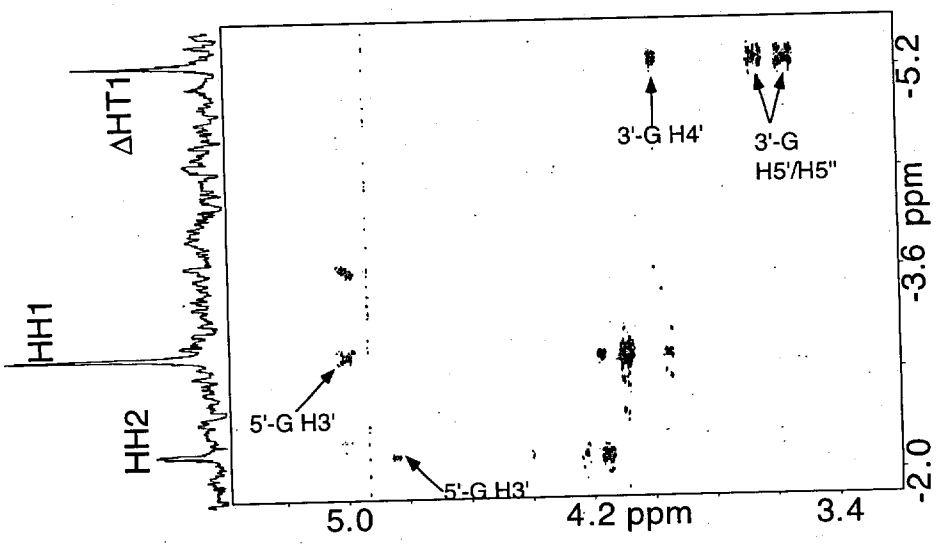

Figure S5. ^{1}H-^{31}P HMBC spectrum of **Me_2ppz**Pt(d(GpG)) collected at 5 °C, pH ~4.

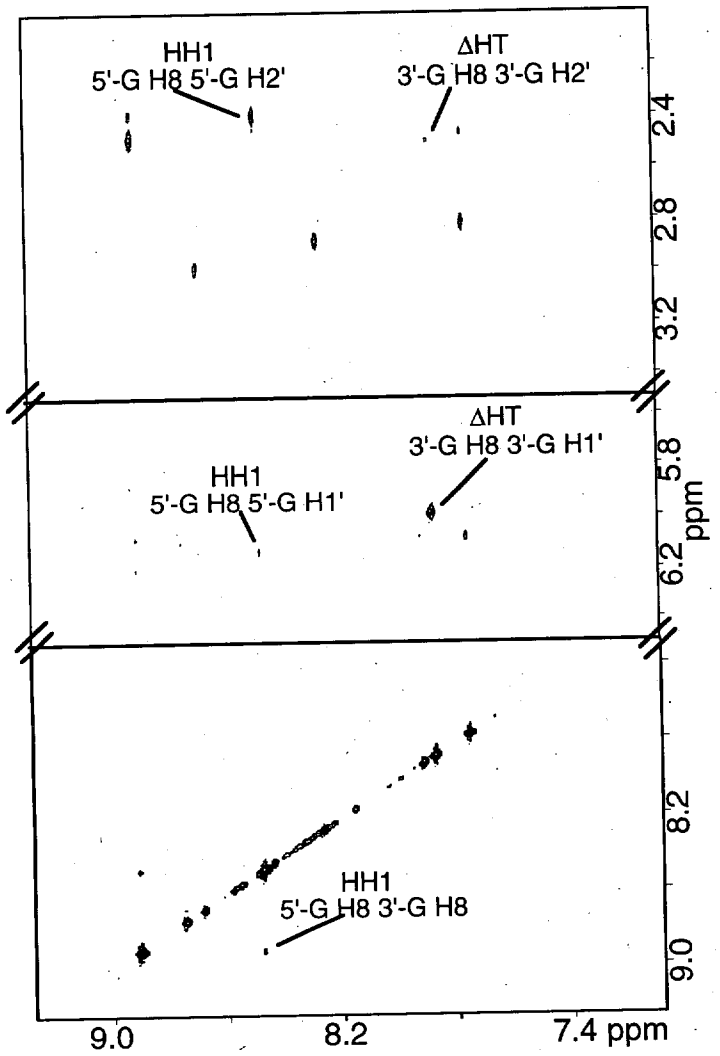

Figure S6. Regions of **Me$_2$ppz**Pt(d(GpG)) ROESY spectrum showing H8-H8 NOE cross-peaks and base-sugar NOE cross-peaks, collected at 5 °C, pH ~4 in D_2O.