

Electronic Supplementary Information

**Synthesis of 2,2'-Biimidazole-Based Platinum(II)
Polymetallaynes and Tuning Their Fluorescent Response
Behaviors to Cu²⁺ Ions Through Optimizing the Configuration
of the Organic Spacers and Steric Effect**

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R. China.

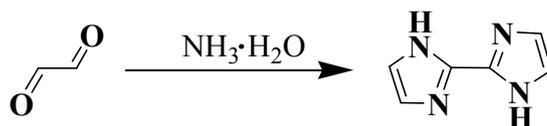
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Synthesis of 2,2'-Biimidazole. To the solution of glyoxal (25 mL, 40%), the concentrate ammonia (30 mL, *ca.* 28%) was added slowly in about 0.5 h. After addition, the reaction was stirred for 5 h. The brown precipitate was collected by filtration and washed with water (20 mL). The crude product was recrystallized with ethylene glycol. The hot ethylene glycol solution was carefully poured into a beaker. After cooling to room temperature, the formed needle crystals were collected and washed with ethanol (30 mL). The 2,2'-biimidazole was obtained in *ca.* 45% yield. ¹H NMR (400 MHz, DMSO-*d*₆): δ (ppm) 12.67 (s, br, 2H), 7.14 (s, 1H), 7.10 (s, 1H).



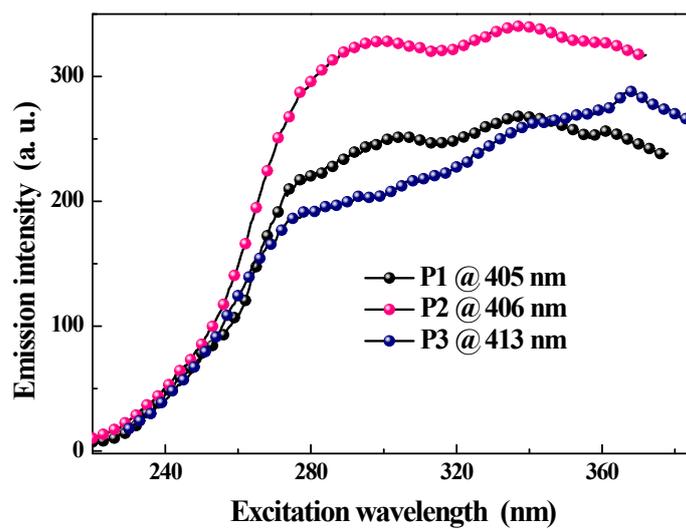


Figure S1. The excitation spectra for these polymetallaynes in THF at 293 K.

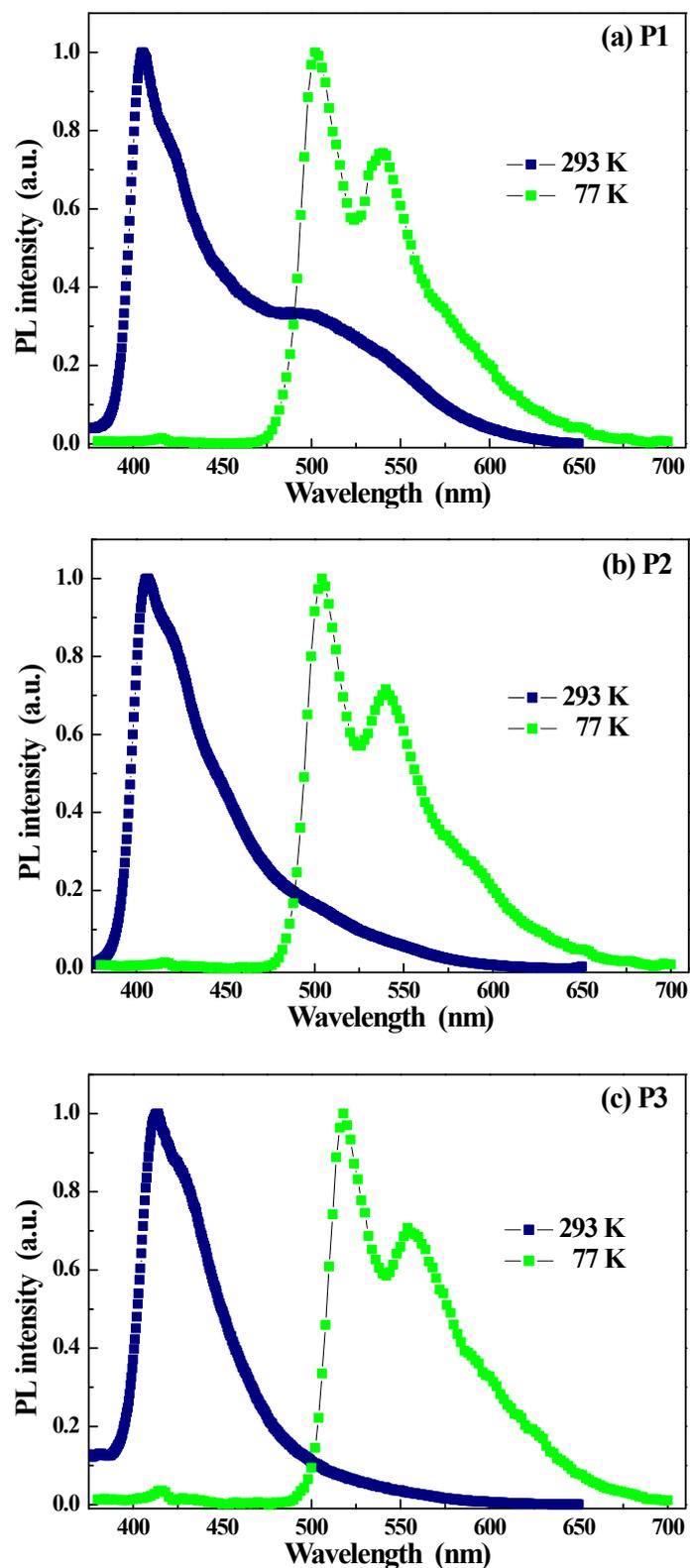


Figure S2. The emission spectra for the 2,2'-biimidazole-based polymetallaynes in THF at 293 K and 77K.

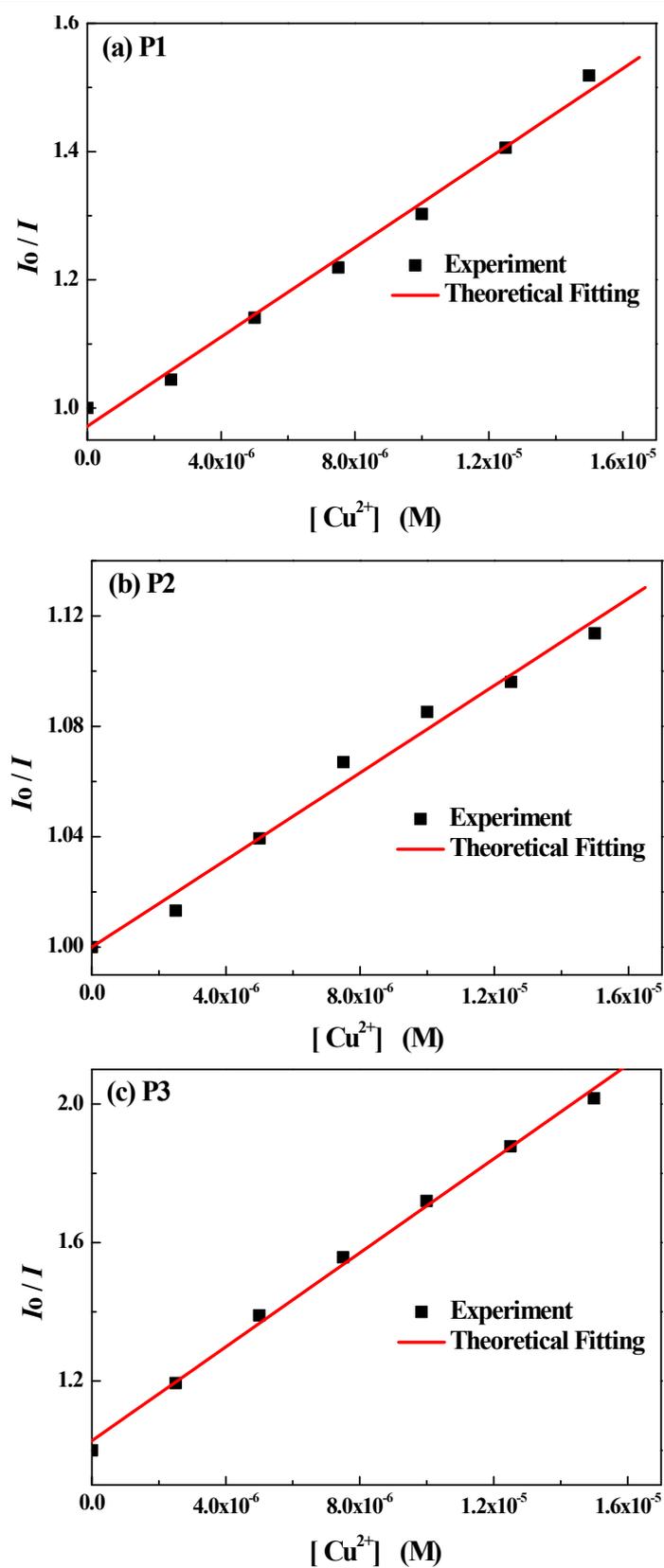


Figure S3. The fitting results for the K_{SV} based on the fluorescence titration results.

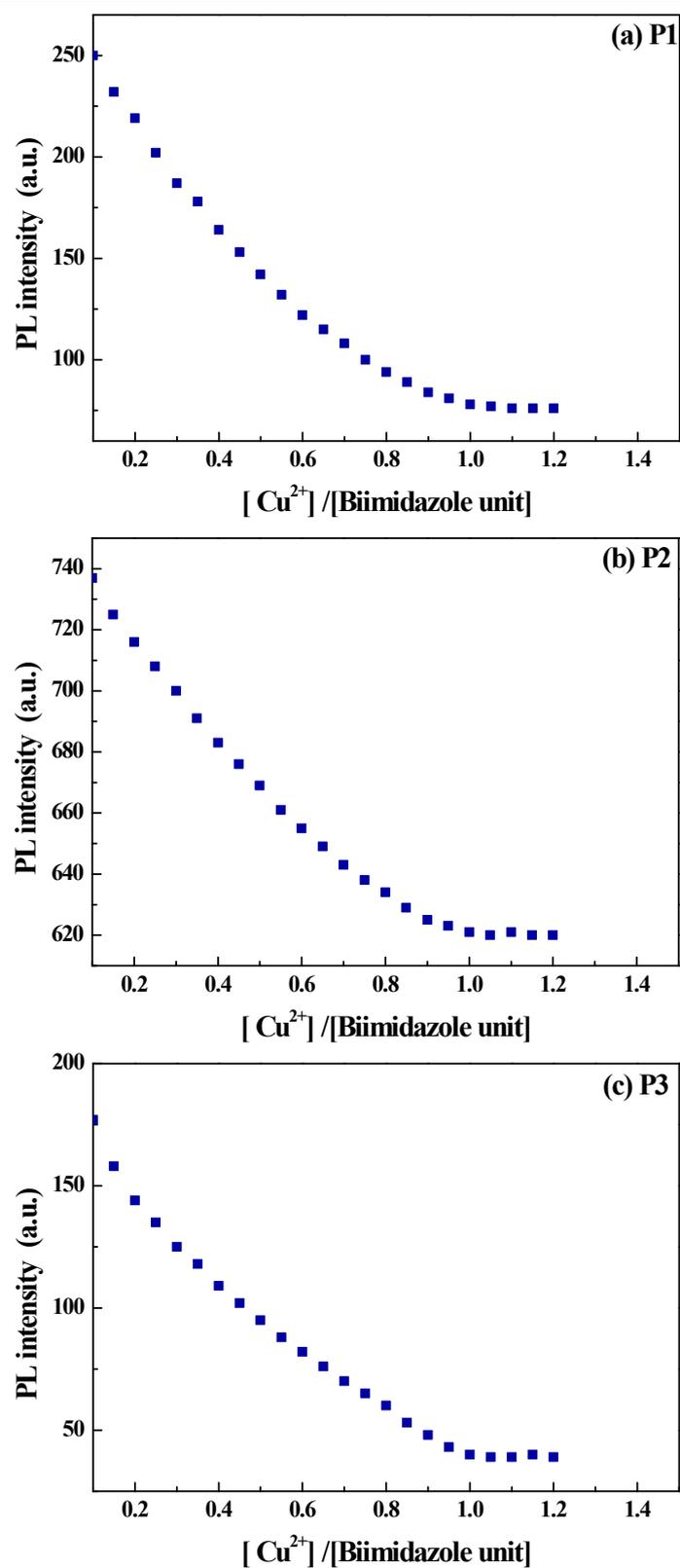


Figure S4. The titration plots for the Cu^{2+} sensing processes of the 2,2'-biimidazole-based polymetallaynes in THF.

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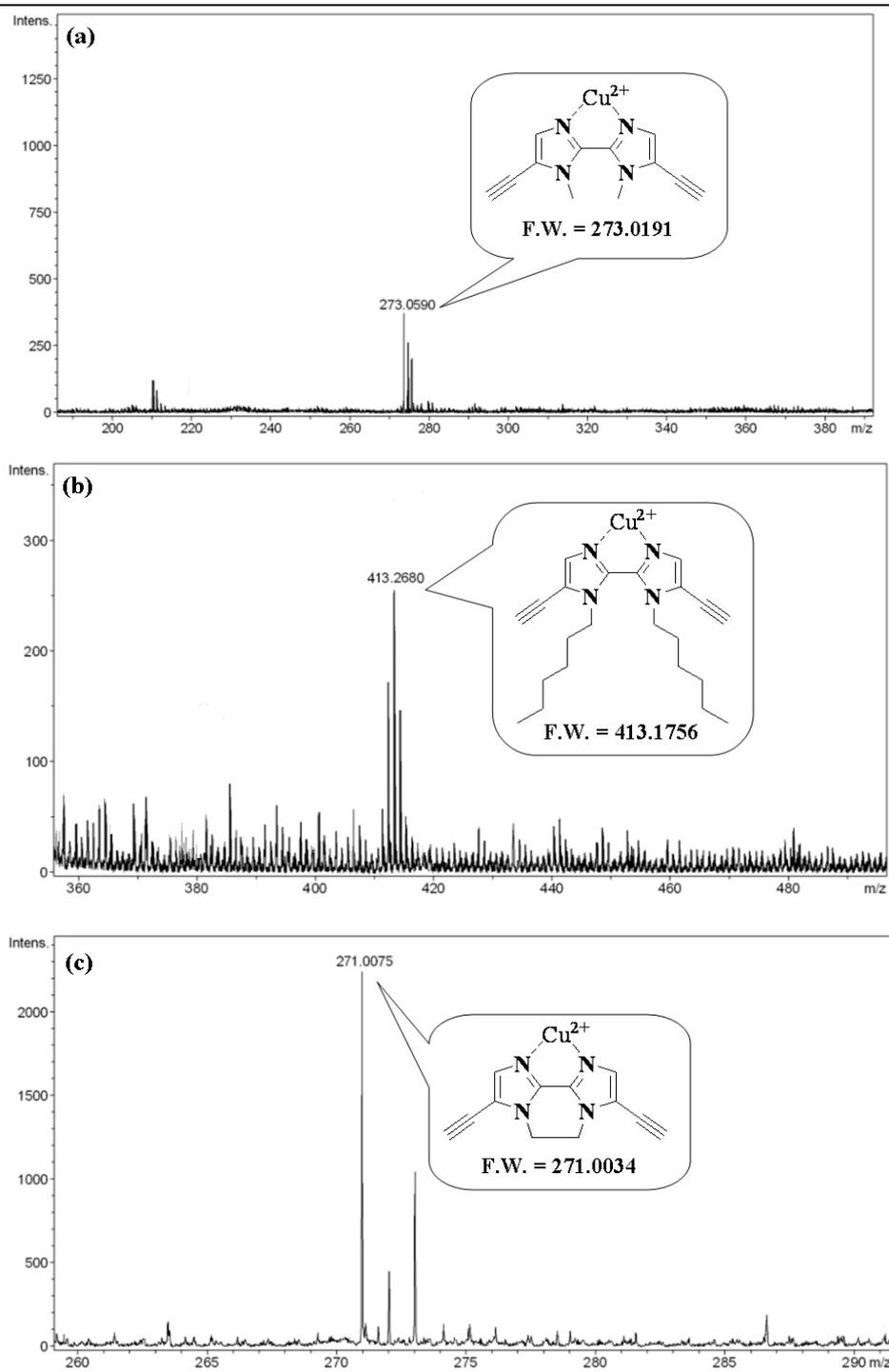


Figure S5. The mass spectra for the possible adducts between the Cu^{2+} ions and organic ligands.

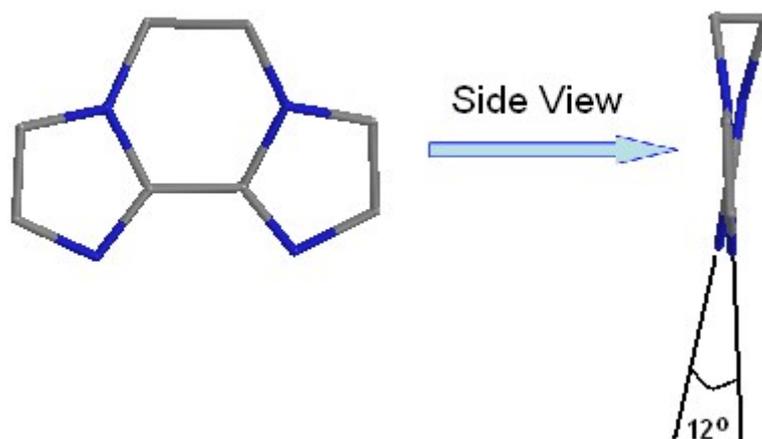


Figure S6. The arrangement of the two imidazole rings in the 2,2'-biimidazole units with optimized configuration in **P3**.

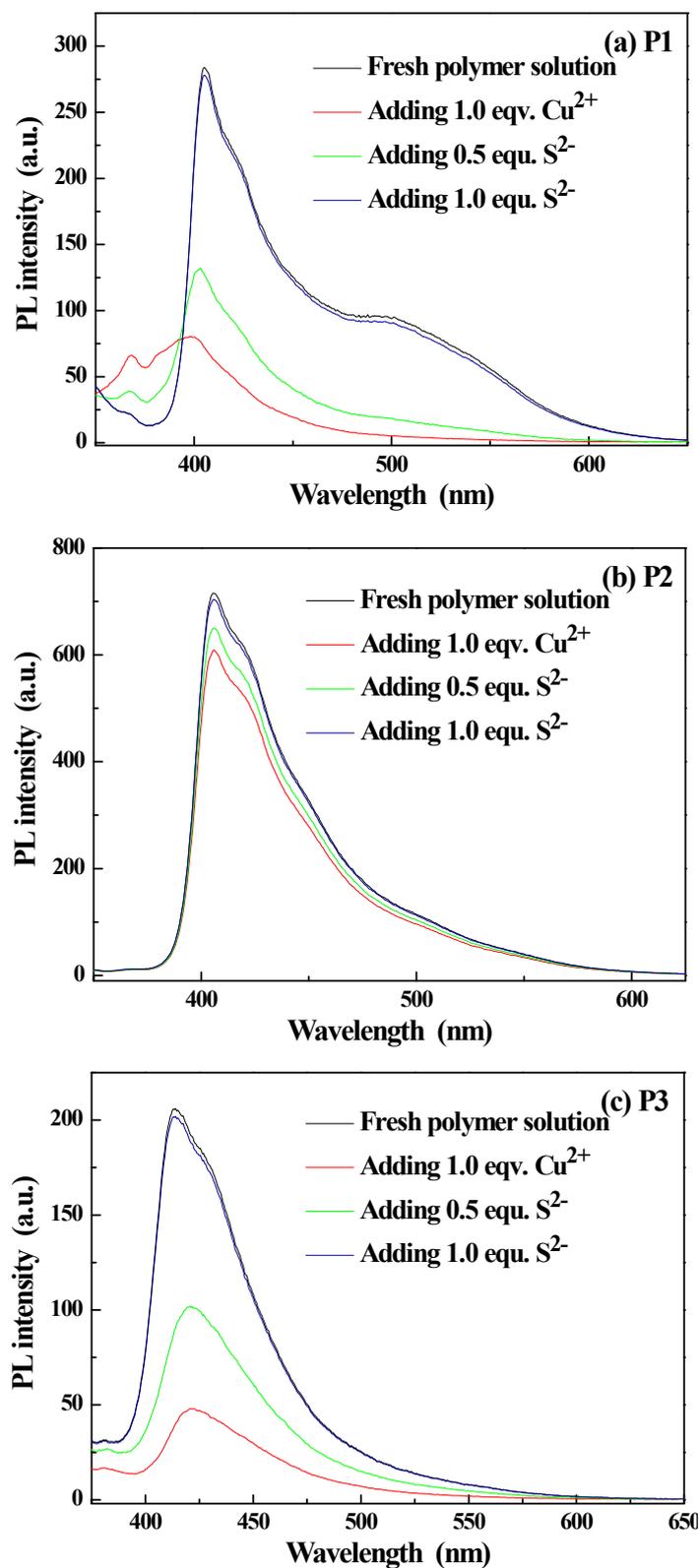


Figure S7. The emission turn-on behaviors by S^{2-} anions of the 2,2'-biimidazole-based polymetallaynes in THF.

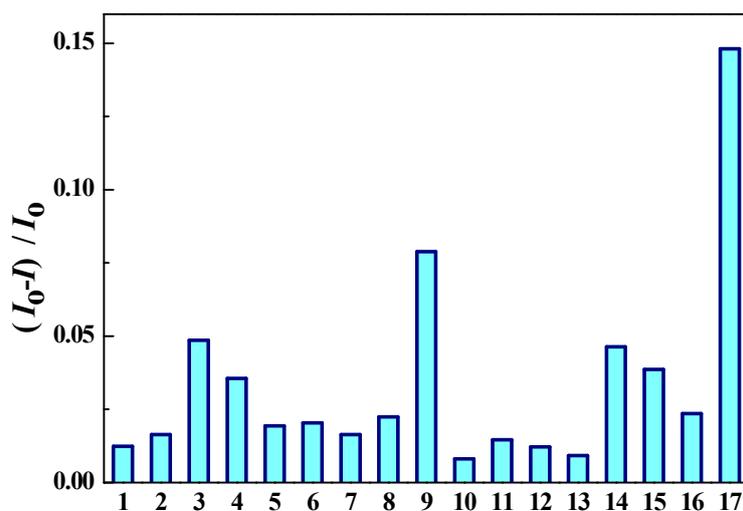


Figure S8. The selectivity of the fluorescent signal of the **P2** to the Cu²⁺ ions (I : The fluorescent signal intensity after adding 1.2 equivalent of different ion; I_0 : The fluorescent signal intensity before adding ions). **1**: Ag⁺, **2**: Zn²⁺, **3**: Fe³⁺, **4**: Ni²⁺, **5**: Na⁺, **6**: Mn²⁺, **7**: Mg²⁺, **8**: K⁺, **9**: Co²⁺, **10**: Hg²⁺, **11**: Cd²⁺, **12**: Ca²⁺, **13**: NH₄⁺, **14**: Ru³⁺, **15**: Ir³⁺, **16**: Rh³⁺, **17**: Cu²⁺.