# Crystal structure of (2,2'-bipyridine-$\kappa^2N,N'$)dibromidopalladium(II) dibromomethane monosolvate, $C_{11}H_{10}Br_4N_2Pd$

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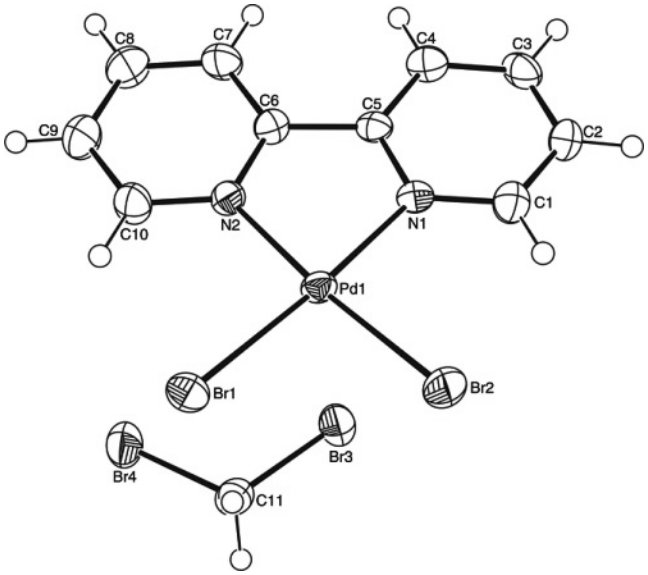
Received January 14, 2014, accepted April 09, 2014, available online April 22, 2014, CCDC no. 1267/4096




**Abstract**

$C_{11}H_{10}Br_4N_2Pd$, triclinic, $P\bar{1}$ (no. 2), $a$ = 9.0574(9) Å, $b$ = 9.4029(9) Å, $c$ = 10.100(1) Å, $\alpha$ = 74.091(2)°, $\beta$ = 66.818(2)°, $\gamma$ = 81.760(2)°, $V$ = 759.9 Å$^3$, $Z$ = 2, $R_{gt}(F)$ = 0.0316, $wR_{ref}(F^2)$ = 0.0866, $T$ = 200 K.

**Table 1.** Data collection and handling.

| | |
|---|---|
| Crystal: | orange blocks, size 0.12×0.15×0.18 mm |
| Wavelength: | Mo $K_\alpha$ radiation (0.71073 Å) |
| $\mu$: | 117.21 cm$^{-1}$ |
| Diffractometer, scan mode: | Bruker SMART 1000 CCD, $\varphi$ and $\omega$ |
| $2\theta_{max}$: | 51.94° |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$: | 4655, 2885 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\ \sigma(I_{obs})$, 2386 |
| $N(param)_{refined}$: | 163 |
| Programs: | SHELX [6], ORTEP-3 [7], PLATON [8] |

## Source of material

To a solution of $K_2PdBr_4$ (0.251 g, 0.50 mmol) in $H_2O$ (20 ml) was added 2,2'-bipyridine (*bipy*; 0.082 g, 0.52 mmol) and stirred for 3 h at room temperature. The formed precipitate was separated by filtration, washed with $H_2O$ and acetone, and dried at 50 °C, to give an orange powder (0.134 g). Crystals suitable for X-ray diffraction analysis were obtained by slow evaporation from a $CH_2Br_2$ solution.

## Experimental details

Hydrogen atoms were positioned geometrically and allowed to ride on their parent atoms with $d$(C–H) = 0.95 Å (CH) or 0.99 Å ($CH_2$) and $U_{iso}$(H) = 1.2$U_{eq}$(C). The highest peak (0.87 e·Å$^{-3}$) and the deepest hole (–0.94 e·Å$^{-3}$) in the difference Fourier map are located 0.85 Å and 0.88 Å from the atoms Br4 and Pd1, respectively.

## Discussion

This contribution is part of my continuing interest in the structural chemistry of Pd-bipyridine complexes [1-5]. The title compound $[PdBr_2(bipy)]\cdot CH_2Br_2$ is isotypic with the previously reported analogous Pd(II) complexes $[PdX_2(bipy)]\cdot CH_2Cl_2$ (X = Cl or Br) [1, 2]. In the neutral complex, the Pd(II) ion is four-coordinated in a slightly distorted square-planar environment by two N atoms of the *bipy* ligand and two bromido ligands. The main contribution to the distortion is the tight N1–Pd1–N2 chelate angle of 80.27(17)°, which results in non-linear *trans* arrangement (<N1–Pd1–Br1 = 175.7(1)° and <N2–Pd1–Br2 = 174.9(1)°). The two Pd–Br bond lengths are nearly equal with $d$(Pd–Br) = 2.4221(7) and 2.4115(7) Å, whereas the Pd–N bond lengths are exactly equivalent within the experimental error with $d$(Pd–N) = 2.043(4) Å. The nearly planar *bipy* ligand (maximum deviation = 0.040(4) Å) is slightly inclined to the $PdBr_2N_2$ coordination plane (maximum deviation = 0.026(1) Å), making dihedral angle of 5.9(2)°. The complex displays weak intramolecular C–H···Br hydrogen bonds with $d$(C···Br) = 3.346(6) and 3.350(6) Å. Pairs of complex molecules are assembled by intermolecular C–H···Br hydrogen bonds with $d$(C···Br) = 3.712(6) Å. These pairs are connected by additional intermolecular hydrogen bonds with $d$(C···Br) = 3.647(6) and 3.685(6) Å, thereby forming a layer structure extending parallel to (011).

**Table 2.** Atomic coordinates and displacement parameters (in Å$^2$).

| Atom | Site | $x$ | $y$ | $z$ | $U_{iso}$ |
|---|---|---|---|---|---|
| H(1) | 2*i* | 0.1296 | 0.5278 | 0.0524 | 0.039 |
| H(2) | 2*i* | 0.1175 | 0.7831 | –0.0226 | 0.040 |
| H(3) | 2*i* | 0.2875 | 0.9199 | 0.0140 | 0.042 |
| H(4) | 2*i* | 0.4634 | 0.7964 | 0.1305 | 0.037 |
| H(7) | 2*i* | 0.6207 | 0.6657 | 0.2380 | 0.044 |
| H(8) | 2*i* | 0.7739 | 0.5116 | 0.3645 | 0.053 |
| H(9) | 2*i* | 0.7383 | 0.2569 | 0.4371 | 0.053 |
| H(10) | 2*i* | 0.5486 | 0.1618 | 0.3866 | 0.041 |
| H(11A) | 2*i* | –0.0572 | 0.0635 | 0.7052 | 0.043 |
| H(11B) | 2*i* | 0.0888 | 0.0585 | 0.5515 | 0.043 |

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**Table 3.** Atomic coordinates and displacement parameters (in Å$^2$).

| Atom | Site | $x$ | $y$ | $z$ | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|---|---|---|---|
| Pd(1) | 2*i* | 0.32164(5) | 0.28585(4) | 0.21127(4) | 0.0220(2) | 0.0203(2) | 0.0287(2) | –0.0034(2) | –0.0096(2) | –0.0067(2) |
| Br(1) | 2*i* | 0.36844(7) | 0.02355(6) | 0.30161(7) | 0.0379(3) | 0.0209(3) | 0.0446(4) | –0.0027(2) | –0.0173(3) | –0.0075(2) |
| Br(2) | 2*i* | 0.14468(8) | 0.22617(7) | 0.10939(8) | 0.0469(4) | 0.0326(3) | 0.0584(4) | –0.0125(3) | –0.0339(3) | –0.0046(3) |
| N(1) | 2*i* | 0.2962(5) | 0.5098(5) | 0.1385(5) | 0.022(2) | 0.024(2) | 0.022(2) | –0.002(2) | –0.005(2) | –0.008(2) |
| N(2) | 2*i* | 0.4759(5) | 0.3533(5) | 0.2826(5) | 0.023(2) | 0.020(2) | 0.033(3) | 0.001(2) | –0.010(2) | –0.008(2) |
| C(1) | 2*i* | 0.1973(7) | 0.5831(6) | 0.0698(6) | 0.031(3) | 0.035(3) | 0.034(3) | 0.002(3) | –0.014(3) | –0.008(3) |
| C(2) | 2*i* | 0.1903(7) | 0.7346(6) | 0.0239(7) | 0.028(3) | 0.033(3) | 0.039(3) | 0.004(2) | –0.017(3) | –0.005(3) |
| C(3) | 2*i* | 0.2898(7) | 0.8150(6) | 0.0460(7) | 0.031(3) | 0.021(3) | 0.040(3) | 0.004(2) | –0.007(3) | 0.001(2) |
| C(4) | 2*i* | 0.3932(7) | 0.7421(6) | 0.1152(6) | 0.031(3) | 0.027(3) | 0.034(3) | –0.003(2) | –0.008(3) | –0.011(2) |
| C(5) | 2*i* | 0.3937(6) | 0.5889(6) | 0.1621(6) | 0.020(3) | 0.021(3) | 0.025(3) | –0.001(2) | –0.002(2) | –0.007(2) |
| C(6) | 2*i* | 0.4964(6) | 0.5014(6) | 0.2403(6) | 0.027(3) | 0.028(3) | 0.024(3) | –0.002(2) | –0.010(2) | –0.009(2) |
| C(7) | 2*i* | 0.6070(8) | 0.5619(7) | 0.2691(7) | 0.042(4) | 0.028(3) | 0.044(4) | –0.010(3) | –0.020(3) | –0.002(3) |
| C(8) | 2*i* | 0.6977(8) | 0.4711(7) | 0.3434(7) | 0.047(4) | 0.043(4) | 0.057(4) | –0.013(3) | –0.031(3) | –0.010(3) |
| C(9) | 2*i* | 0.6764(8) | 0.3213(7) | 0.3865(7) | 0.044(4) | 0.046(4) | 0.051(4) | –0.006(3) | –0.031(3) | –0.004(3) |
| C(10) | 2*i* | 0.5640(7) | 0.2653(6) | 0.3555(7) | 0.040(4) | 0.024(3) | 0.044(4) | –0.001(3) | –0.023(3) | –0.008(3) |
| Br(3) | 2*i* | –0.00667(8) | 0.31497(7) | 0.55064(7) | 0.0382(4) | 0.0386(4) | 0.0407(4) | 0.0013(3) | –0.0172(3) | –0.0014(3) |
| Br(4) | 2*i* | 0.19293(9) | 0.09673(8) | 0.72652(9) | 0.0576(5) | 0.0401(4) | 0.0777(5) | –0.0028(3) | –0.0475(4) | –0.0020(3) |
| C(11) | 2*i* | 0.0430(7) | 0.1116(6) | 0.6320(7) | 0.029(3) | 0.031(3) | 0.049(4) | –0.001(2) | –0.015(3) | –0.009(3) |

*Acknowledgments.* This work was supported by Priority Research Centers Program through the National Research Foundation of Korea (NRF) funded by the Ministry of Education, Science and Technology (2009-0094055). The author thanks the KBSI, Jeonju Center, for the X-ray data collection.

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