X-ray powder diffraction data for a PicoPtBr$_2$, *cis*-amminedibromo (2-methylpyridine)platinum complex [Pt(NH$_3$)(2-pic)Br$_2$]

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X-ray powder diffraction data, unit-cell parameters, and space group for a novel platinum-based anticancer complex, Pt($C_6H_{10}N_2$)Br$_2$, are presented [a = 10.049 (1) Å, b = 9.240 (2) Å, c = 11.497 (2) Å, β = 101.945 (3)°, unit-cell volume V = 1044.41 Å^3, Z = 4, and space group $P2_1/c$]. All the measured lines were indexed and are consistent with the $P2_1/c$ space group. No detectable impurities were observed. © *2012 International Centre for Diffraction Data.*
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Key words: X-ray powder diffraction, crystal structure, cytostatics, PicoPtBr$_2$

I. INTRODUCTION

Cancer is a class of diseases which accounts for approximately 13% of all deaths each year worldwide. Cancer is thought to be caused by the interaction between genetic susceptibility and environmental factors. Chemotherapeutic drugs (=Cytostatics) are often used as a part of the complex anticancer therapy. Their mechanism of action consists in impairing mitosis (cell division) and effectively targeting fast-dividing cancer cells. Platinum complexes represent a significant group of these compounds.

Cis-amminedibromo(2-methylpyridine)platinum complex [Pt(NH$_3$)(2-pic)Br$_2$] (herein referred to as PicoPtBr$_2$) is an analogue of Picoplatin (amminedichloro (2-methylpyridine)platinum complex), which is currently used clinically. PicoPtBr$_2$ represents an analogue that showed a similar cytostatic activity but slightly different pharmakokinetic behaviour caused by distinct water solubility and thus potentially different side effects.

We have inspected the CSD database (Allen, 2002) for similar compounds and found the crystal data for the isostructural analogue Picoplatin Pt($C_6H_{10}N_2$)Cl$_2$ (Battle *et al.*, 2006). This compound is monoclinic [a = 9.878(1) Å, b = 9.0223(9) Å, c = 11.202(1) Å, β = 102.595(2)°, unit-cell volume V = 973.32(1) Å^3, Z = 4, and space group $P2_1/c$]. The calculated powder pattern of this compound is similar to the measured powder pattern of the title compound reported in this paper.

II. SAMPLE PREPARATION

Preparation of PicoPtBr$_2$ (Figure 1) is based on the principle of synthesis of platinum complexes with diamincyclohexane ligand, which was published by Schwartz *et al.* (1977) and Pasini *et al.* (1993).

Starting compounds for synthesis were Picoplatin [amminedichloro(2-methylpyridine) platinum complex] and AgNO$_3$ to form amminedinitro(2-methylpyridine)platinum complex. This substance reacted with KBr to form an amminedibromo (2-methylpyridine)platinum complex in the second reaction step.

III. POWDER DIFFRACTION DATA

The diffraction pattern for the title compound was collected at room temperature with an X'Pert PRO θ–θ powder diffractometer with parafocusing Bragg–Brentano geometry using Cu$K\alpha_1$ radiation (λ = 1.5406 Å, Ni filter, generator setting: 40 kV, 30 mA). An ultrafast X'Celerator detector was employed to collect XRD data over the angular range from 4 to 70°2θ with a step size of 0.017°2θ and a counting time of 81.28 s per step. The experimental powder diffraction pattern is depicted in Figure 2. The software package HighScore Plus V3.0e(PANalytical, Almelo, The Netherlands) was used to smooth the data, to fit the background, to eliminate the $K\alpha_2$ component, and the top of smoothed peak method was used to determine the peak positions and intensities of the diffraction peaks (Table 1).

The collected data are consistent with monoclinic unit-cell parameters [a = 10.049 (1) Å, b = 9.240 (2) Å, c = 11.497 (2) Å, β = 101.945 (3)°, unit-cell volume V = 1044.41 Å^3, Z = 4, and space group $P2_1/c$]. These parameters were derived using DICVOL04 (Boultif and Louër, 2004) with the results all being within the errors indicated. The following figures of merit were achieved F_{20} = 85.6 (0.0057 41) (Smith and Snyder, 1979) and M_{20} = 37.8 (de Wolff, 1968).

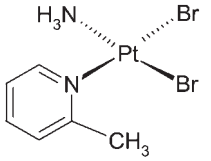


Figure 1. Structural formula of Pt($C_6H_{10}N_2$)Br$_2$.

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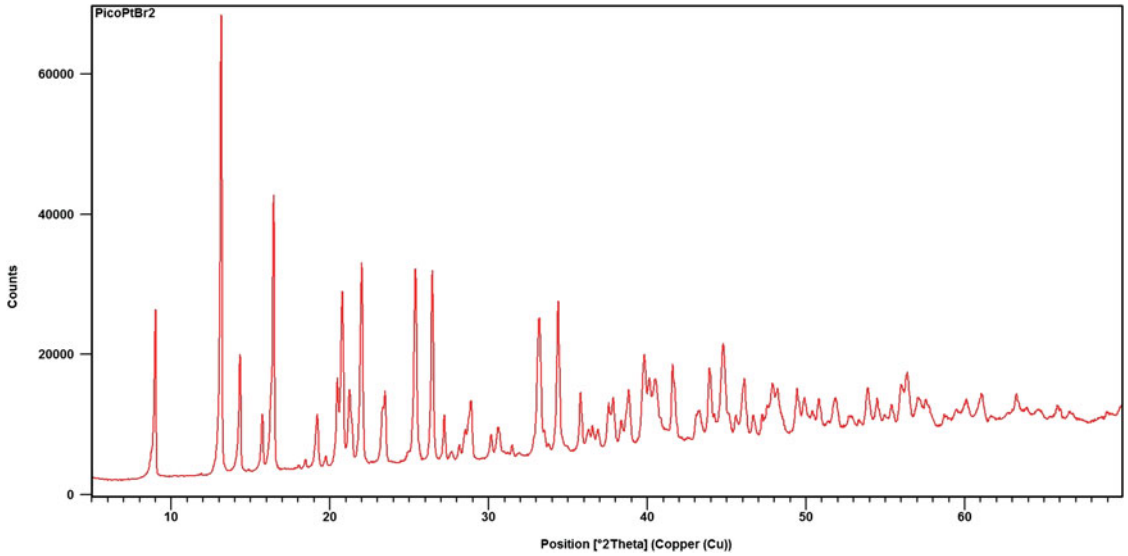


Figure 2. X-ray powder diffraction pattern of $Pt(C_6H_{10}N_2)Br_2$ using Cu$K\alpha_1$ radiation ($\lambda = 1.5406$ Å).

TABLE I. Indexed X-ray powder diffraction data for $Pt(C_6H_{10}N_2)Br_2$. Only the peaks with I_{rel} of 1 or greater are presented [$a = 10.049$ (1) Å, $b = 9.240$ (2) Å, $c = 11.497$ (2) Å, $\beta = 101.945$ (3)°, unit-cell volume $V = 1044.41$ Å^3, $Z = 4$, and space group $P2_1/c$]. All lines were indexed and are consistent with the $P2_1/c$ space group. The d-values were calculated using Cu$K\alpha_1$ radiation ($\lambda = 1.5406$ Å).

$2\theta_{obs}$ (°)	d_{obs} (Å)	I_{obs}	h	k	l	$2\theta_{cal}$ (°)	d_{calc} (Å)	$\Delta 2\theta$
8.987	9.821	36	1	0	0	8.988	9.831	−0.009
13.147	6.729	100	1	1	0	13.139	6.733	−0.009
14.332	6.175	25	1	1	−1	14.330	6.176	−0.001
15.737	5.627	12	0	0	2	15.745	5.624	0.008
16.276	5.442	15	1	1	1	16.260	5.447	−0.016
16.444	5.386	60	1	0	−2	16.446	5.386	0.002
18.032	4.916	1	2	0	0	18.031	4.916	0.000
18.459	4.803	2	0	1	2	18.454	4.804	−0.006
19.197	4.620	12	0	2	0	19.195	4.620	−0.002
19.733	4.495	2	1	0	2	19.726	4.497	−0.007
20.468	4.336	19	2	1	0	20.448	4.340	−0.020
20.771	4.273	38	0	2	−1	20.768	4.274	−0.003
21.232	4.181	16	1	2	0	21.231	4.181	−0.001
21.389	4.151	9	2	0	−2	21.387	4.151	−0.002
21.993	4.038	43	1	2	−1	21.999	4.037	0.006
23.325	3.811	11	1	2	1	23.321	3.811	−0.005
23.469	3.788	16	2	1	−2	23.474	3.787	0.005
24.946	3.567	2	0	2	2	24.922	3.570	−0.024
25.386	3.506	42	1	2	−2	25.379	3.507	−0.007
26.445	3.368	41	2	2	0	26.454	3.367	0.009
27.206	3.275	10	3	0	0	27.190	3.277	−0.017
27.656	3.223	2	1	2	2	27.660	3.222	0.004
28.154	3.167	3	2	1	2	28.152	3.167	−0.002
28.520	3.127	6	3	0	−2	28.522	3.127	0.003
28.740	3.104	8	2	2	1	28.714	3.106	−0.026
28.878	3.089	12	3	1	0	28.884	3.089	0.005
30.150	2.962	5	3	1	−2	30.148	2.962	−0.002
30.604	2.919	6	1	2	−3	30.567	2.922	−0.037
30.705	2.909	5	0	2	3	30.685	2.911	−0.020
30.946	2.887	1	1	3	−1	30.941	2.888	−0.005
31.483	2.839	2	3	1	1	31.472	2.840	−0.011
33.187	2.697	28	0	3	2	33.135	2.701	−0.052
33.502	2.673	5	3	2	0	33.498	2.673	−0.004
33.778	2.651	2	3	1	−3	33.782	2.651	0.004
34.384	2.606	33	2	3	−1	34.381	2.606	−0.003
34.947	2.565	1	1	0	4	34.928	2.567	−0.018
35.794	2.507	13	3	2	1	35.787	2.507	−0.007
36.286	2.474	4	2	3	−2	36.289	2.474	0.002
36.547	2.457	5	4	0	0	36.529	2.458	−0.018
36.883	2.435	4	1	2	−4	36.888	2.435	0.005
37.561	2.393	9	3	0	−4	37.559	2.393	−0.002
37.845	2.375	11	4	1	0	37.847	2.375	0.001
38.361	2.345	6	2	2	3	38.352	2.345	−0.009
38.697	2.325	8	2	2	−4	38.671	2.327	−0.027
38.824	2.318	13	3	1	−4	38.847	2.316	0.023
39.806	2.263	20	0	4	1	39.804	2.263	−0.001
40.132	2.245	15	3	3	0	40.146	2.244	0.014
40.502	2.225	14	1	4	−1	40.500	2.226	−0.002
40.877	2.206	6	4	2	−1	40.857	2.207	−0.020
41.595	2.169	18	4	2	0	41.586	2.170	−0.009
41.703	2.164	13	2	1	−5	41.677	2.165	−0.026
42.562	2.122	1	1	4	−2	42.549	2.123	−0.013
43.086	2.098	6	1	3	−4	43.087	2.098	0.001
43.216	2.092	7	2	4	0	43.239	2.091	0.023
43.297	2.088	7	2	4	−1	43.280	2.089	−0.017
43.915	2.060	16	3	3	−3	43.948	2.059	0.033
43.992	2.057	14	1	2	−5	43.959	2.058	−0.032
44.227	2.046	6	4	1	2	44.226	2.046	−0.001
44.779	2.022	21	0	2	−5	44.773	2.023	−0.005
45.144	2.007	6	2	2	−5	45.153	2.006	0.009
45.573	1.989	6	5	0	−2	45.576	1.989	0.004
46.101	1.967	13	0	4	3	46.117	1.967	0.015
46.679	1.944	5	3	0	4	46.677	1.944	−0.001
47.236	1.923	5	5	1	0	47.222	1.923	−0.013
47.537	1.911	8	5	0	−3	47.525	1.912	−0.012
47.892	1.898	12	2	4	−3	47.876	1.898	−0.016
48.206	1.886	11	2	0	−6	48.202	1.886	−0.004
49.437	1.842	11	5	2	−1	49.437	1.842	0.000
49.894	1.826	8	5	2	−2	49.882	1.827	−0.012
50.390	1.809	6	5	2	0	50.397	1.809	0.008
50.797	1.796	8	5	0	−4	50.816	1.795	0.018
51.378	1.777	3	1	0	6	51.437	1.775	0.059
51.844	1.762	8	3	1	−6	51.839	1.762	−0.005
52.698	1.736	4	1	3	5	52.705	1.735	0.007
52.915	1.729	4	3	4	2	52.919	1.729	0.004
53.309	1.717	3	1	4	4	53.310	1.717	0.001
53.887	1.700	10	4	4	−1	53.874	1.700	−0.013
54.496	1.682	7	5	3	−1	54.494	1.683	−0.003
54.958	1.669	4	3	1	5	54.938	1.670	−0.020

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TABLE I. Continued

$2\theta_{obs}$ (°)	d_{obs} (Å)	I_{obs}	h	k	l	$2\theta_{cal}$ (°)	d_{calc} (Å)	$\Delta 2\theta$
55.380	1.658	6	0	5	3	55.382	1.658	0.002
56.008	1.641	10	1	0	−7	56.000	1.641	−0.008
56.362	1.631	13	4	4	1	56.357	1.631	−0.005
57.053	1.613	7	6	1	0	57.037	1.613	−0.016
57.546	1.600	7	5	3	1	57.563	1.600	0.017
58.696	1.572	3	6	2	−1	58.689	1.572	−0.006
59.459	1.553	4	4	4	2	59.462	1.553	0.003
60.113	1.538	6	1	3	6	60.113	1.538	0.000
61.052	1.517	7	5	4	−1	61.086	1.516	0.034
61.659	1.503	2	5	1	−6	61.658	1.503	−0.001
62.663	1.481	3	6	2	−4	62.681	1.481	0.019
62.982	1.475	4	1	4	−6	62.980	1.475	−0.002
63.248	1.469	7	6	3	−1	63.245	1.469	−0.003
63.897	1.456	4	0	4	−6	63.901	1.456	0.003
64.578	1.442	3	2	0	7	64.592	1.442	0.014
65.865	1.417	4	1	1	−8	65.856	1.417	−0.009
66.051	1.413	3	7	1	−1	66.033	1.414	−0.018
66.613	1.403	3	6	3	1	66.617	1.403	0.005
68.614	1.367	1	7	2	−1	68.623	1.367	0.009
68.969	1.361	2	7	1	−4	68.974	1.360	0.005

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