X-ray powder diffraction data for [amminedichloro(3,5-dimethyl-tricyclo [3.3.1.1^{3,7}]decan-1-amine)-platinum (II) complex

R. Pažout,[1,a] J. Maixner,[1] J. Svoboda,[2] and P. Kačer[2]
[1]*Central Laboratories, University of Chemical Technology (UCT) Prague, Technická 5, 166 28 Prague 6, Czech Republic*
[2]*Department of Organic Technology, University of Chemical Technology Prague, Technická 5, 166 28 Prague, Czech Republic*

(Received 5 July 2015; accepted 27 September 2015)

X-ray powder diffraction data, unit-cell parameters, and space group for a new memantine analogue of a Platinum (Pt) (II) complex labelled LA-13, $C_{12}H_{24}Cl_2N_2Pt$, are reported [$a = 8.324(1)$ Å, $b = 27.838(2)$ Å, $c = 7.113(1)$, $\beta = 111.25(1)$, unit-cell volume $V = 1536.26$ Å^3, $Z = 4$, and space group $P2_1/n$]. All measured lines were indexed and are consistent with the $P2_1/n$ space group. No detectable impurity was observed. © *2016 International Centre for Diffraction Data*.
[doi:10.1017/S088571561500086X]

Key words: unit cell parameters, space group, X-ray powder diffraction data, memantine Pt (II) complex

I. INTRODUCTION

The platinum (Pt) cytostatics are one of the most frequently used medications for the treatment of cancer. The first Pt cytostatic was based on Pt complexes with oxidation stage (II) and was called cisplatin *cis*–diaminodichloro-platinum (Matthew and Trevor, 2002). This cytostatic has a high cytotoxicity but on the other hand it has many side effects as neurotoxicity, nephrotoxicity, etc. (Galanski *et al.*, 2003). A new strategy for the development of Pt complexes is represented by a new group of complexes where Pt atom has oxidation stage (IV). The first Pt (IV) cytostatic was a complex called satraplatin, [bis (acetato-κO)amminedichloro(cyclohexanamine)-platinum (IV)], followed by the complex LA-12 (Matthew and Trevor, 2002). These complexes have no side effects like previous complexes and they show even more effective cytotoxicity. The Pt complex LA-13 [amminedichloro(3,5-dimethyl-tricyclo[3.3.1.1^{3,7}] decan-1-amine)-platinum(II))] is the first intermediate in the whole synthesis of Pt complex LA-15, [bis(acetato-κO) amminedichloro (3,5-dimethyl tricyclo[3.3.1.1^{3,7}]decan-1-amine)-platinum (IV)], which is a structural analogue of the complex LA-12 as a potential cytostatic.

II. EXPERIMENTAL

A. Sample preparation

The preparation of the title compound is based on the synthesis of LA-12 described in the patent of Žák *et al.* (1999). The synthesis consists of the condensation of the Pt precursor called T-CAP (potassium amminetrichloroplatinate(II)) with the bulky amine ligand memantine (3,5-dimethyladamantane-1-amine). The memantine is then dissolved in methanol and the T-CAP is dissolved in DMF. Subsequently, one molar ratio of T-CAP was mixed with two molar ratios of memantine. The reaction was stirred and heated to 320 K for about 4 h. The product of synthesis (title compound, Figure 1) was filtrated and dried in a vacuum dryer.

B. Diffraction data collection and reduction

The diffraction pattern for the title compound was collected at room temperature using an X'Pert PRO θ-θ powder diffractometer with parafocusing Bragg–Brentano geometry and Cu $K\alpha_1$ radiation ($\lambda = 1.5406$ Å, generator setting: 40 kV, 30 mA). An ultrafast X'Celerator detector was employed to collect X-ray diffraction (XRD) data over the angular range from 4° to 60°2θ with a step size of 0.017°2θ and a counting time of 20.32 s.step^{-1}. The software package HighScore Plus V 3.0d of PANalytical, Almelo, Netherlands, was used to smooth the data, to fit the background and to eliminate the $K\alpha_2$ component. The top of smoothed peak method was used to determine the peak positions and intensities of the diffraction peaks.

Automatic indexing of the experimental XRD pattern was done using DICVOL04 (Boultif and Louër, 2004).

III. RESULTS

The experimental powder diffraction pattern is depicted in Figure 2. Automatic indexing results obtained by DICVOL04 (Table 1) show that the title compound is monoclinic with the

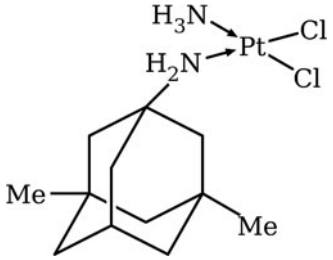

Figure 1. Structural formula of the title amminedichloro(3.5-dimethyl-tricyclo[3.3.1.1$^{3.7}$]decan-1-amine)-platinum (IV) complex.

[a]Author to whom correspondence should be addressed. Electronic mail: richard.pazout@vscht.cz

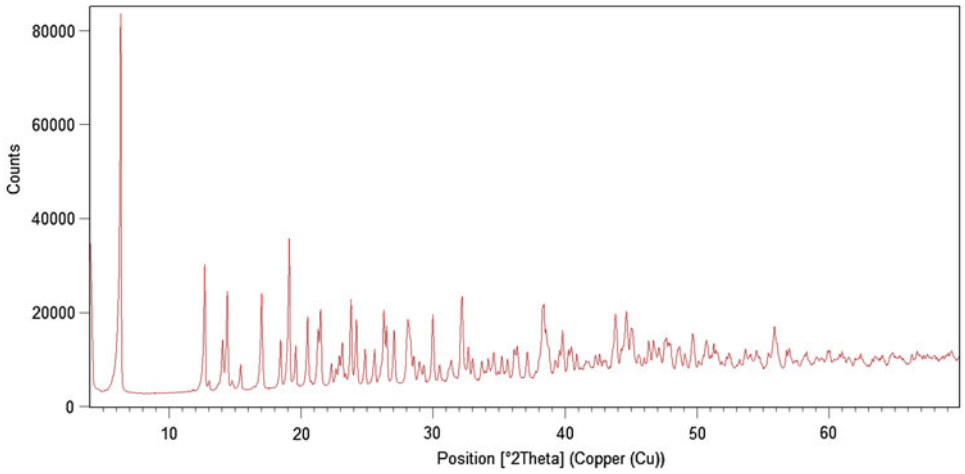

Figure 2. (Color online) X-ray powder diffraction pattern of the title compound using and Cu $K\alpha_1$ radiation ($\lambda = 1.5406$ Å).

TABLE I. Indexed X-ray powder diffraction data for the title compound. $C_{12}H_{24}Cl_2N_2Pt$. Only the peaks with I_{rel} of 1 or greater are presented [$a = 8.324(1)$ Å, $b = 27.838(2)$ Å, $c = 7.113(1)$, $\beta = 111.25(1)$, unit-cell volume $V = 1536.26$ Å^3, $Z = 4$, and space group $P2_1/n$]. All measured lines were indexed and are consistent with the $P2_1/n$ 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)	$\Delta 2\theta$
6.322	13.969	100	0	2	0	6.345	13.919	0.022
11.808	7.489	1	1	1	0	11.833	7.473	0.025
12.689	6.971	33	0	4	0	12.709	6.960	0.020
13.032	6.788	3	1	2	0	13.054	6.776	0.022
13.719	6.450	2	0	1	1	13.719	6.449	0.000
14.049	6.299	13	1	0	−1	14.068	6.290	0.019
14.404	6.144	27	1	1	−1	14.424	6.136	0.020
14.769	5.993	3	0	2	−1	14.789	5.985	0.019
15.423	5.740	7	1	2	−1	15.446	5.732	0.022
17.002	5.211	25	1	3	−1	17.015	5.207	0.014
18.448	4.806	13	0	4	1	18.468	4.800	0.021
19.095	4.644	39	0	6	0	19.113	4.640	0.018
19.593	4.527	11	1	5	0	19.610	4.523	0.017
20.495	4.330	18	1	0	1	20.519	4.325	0.024
20.765	4.274	2	1	1	1	20.768	4.274	0.003
21.289	4.170	15	1	5	−1	21.294	4.169	0.006
21.474	4.135	20	1	2	1	21.498	4.130	0.024
22.295	3.984	6	1	6	0	22.308	3.982	0.013
22.648	3.923	4	1	3	1	22.665	3.920	0.017
22.895	3.881	8	2	0	0	22.909	3.879	0.014
23.118	3.844	11	2	1	0	23.133	3.842	0.015
23.363	3.805	3	0	6	1	23.383	3.801	0.020
23.783	3.738	23	2	2	0	23.794	3.737	0.012
24.189	3.676	17	1	4	1	24.209	3.673	0.021
24.845	3.581	9	2	3	0	24.859	3.579	0.014
25.563	3.482	9	0	8	0	25.578	3.480	0.015
26.063	3.416	5	1	5	1	26.068	3.416	0.005
26.270	3.390	19	2	4	0	26.282	3.388	0.012
26.480	3.363	15	1	7	−1	26.495	3.361	0.014
27.045	3.294	14	0	1	−2	27.068	3.292	0.023
28.086	3.175	17	1	8	0	28.081	3.175	−0.004
28.160	3.166	15	1	6	1	28.185	3.164	0.025
28.246	3.157	12	1	4	−2	28.208	3.161	−0.037
28.541	3.125	7	2	1	−2	28.538	3.125	−0.003
28.949	3.082	5	0	8	−1	28.955	3.081	0.006
29.296	3.046	4	1	8	−1	29.307	3.045	0.011
29.984	2.978	18	2	3	−2	29.974	2.979	−0.010
30.496	2.929	4	1	7	1	30.512	2.927	0.016
31.195	2.865	3	2	4	−2	31.181	2.866	−0.014
31.367	2.850	5	0	5	2	31.382	2.848	0.015
32.193	2.778	22	2	7	0	32.211	2.777	0.018

Continued

https://doi.org/10.1017/S088571561500086X Published online by Cambridge University Press

TABLE I. Continued

$2\theta_{obs}$ (°)	d_{obs} (Å)	I_{obs}	h	k	l	$2\theta_{cal}$ (°)	d_{calc} (Å)	$\Delta 2\theta$
32.667	2.739	9	2	5	−2	32.674	2.738	0.007
32.997	2.712	6	1	8	1	33.012	2.711	0.015
33.697	2.658	5	3	3	−1	33.702	2.657	0.005
34.185	2.621	5	1	10	0	34.193	2.620	0.008
34.593	2.591	7	2	8	0	34.601	2.590	0.008
34.917	2.568	2	0	10	−1	34.928	2.567	0.011
35.206	2.547	6	0	7	2	35.218	2.546	0.012
35.642	2.517	5	1	9	1	35.657	2.516	0.015
36.155	2.482	8	3	5	−1	36.154	2.482	−0.001
36.374	2.468	8	2	7	−2	36.390	2.467	0.015
37.138	2.419	7	2	9	0	37.147	2.418	0.009
37.780	2.379	2	3	4	−2	37.777	2.379	−0.002
38.019	2.365	5	0	11	−1	38.028	2.364	0.009
38.308	2.348	19	1	11	−1	38.305	2.348	−0.003
38.397	2.342	19	1	6	2	38.399	2.342	0.003
38.536	2.334	13	2	8	−2	38.553	2.333	0.017
38.752	2.322	8	0	12	0	38.786	2.320	0.034
39.268	2.292	4	2	10	−1	39.245	2.294	−0.024
39.585	2.275	7	3	7	−1	39.585	2.275	−0.001
39.815	2.262	12	2	10	0	39.826	2.262	0.010
40.273	2.238	7	1	4	−3	40.274	2.238	0.001
40.542	2.223	6	1	12	0	40.556	2.223	0.014
40.892	2.205	5	2	9	−2	40.887	2.205	−0.005
41.185	2.190	2	1	10	−2	41.183	2.190	−0.002
41.481	2.175	3	1	5	−3	41.481	2.175	0.000
41.604	2.169	4	3	8	−1	41.607	2.169	0.003
41.766	2.161	3	2	0	2	41.736	2.162	−0.030
42.318	2.134	5	3	2	1	42.334	2.133	0.016
42.612	2.120	5	2	11	0	42.622	2.120	0.010
42.903	2.106	4	0	4	3	42.904	2.106	0.001
43.078	2.098	4	3	0	−3	43.107	2.097	0.029
43.630	2.073	6	3	2	−3	43.618	2.073	−0.012
43.816	2.065	16	1	13	0	43.822	2.064	0.006
44.261	2.045	6	1	12	1	44.271	2.044	0.010
44.652	2.028	16	1	13	−1	44.666	2.027	0.014
45.049	2.011	12	0	11	−2	45.032	2.012	−0.017
45.576	1.989	4	4	4	−1	45.580	1.989	0.003
45.993	1.972	3	2	11	−2	45.993	1.972	0.000
46.343	1.958	8	3	6	1	46.352	1.957	0.009
46.701	1.943	8	4	4	−2	46.700	1.944	−0.001
47.119	1.927	6	2	8	−3	47.118	1.927	−0.001
47.570	1.910	8	3	6	−3	47.550	1.911	−0.021
47.674	1.906	8	1	3	3	47.676	1.906	0.003
47.845	1.900	7	2	7	2	47.841	1.900	−0.004
47.969	1.895	7	3	10	0	47.979	1.895	0.010
48.530	1.874	5	2	13	0	48.522	1.875	−0.009
48.663	1.870	6	3	11	−1	48.667	1.869	0.004
49.075	1.855	4	3	7	−3	49.077	1.855	0.002
49.681	1.834	9	1	13	−2	49.688	1.833	0.007
50.135	1.818	2	4	1	−3	50.156	1.817	0.021
50.486	1.806	3	1	14	1	50.474	1.807	−0.011
50.717	1.799	8	0	13	−2	50.713	1.799	−0.004
51.048	1.788	4	0	15	−1	51.063	1.787	0.015
51.280	1.780	7	1	15	−1	51.283	1.780	0.003
51.437	1.775	5	2	0	−4	51.469	1.774	0.032
51.592	1.770	4	2	13	−2	51.593	1.770	0.001
51.901	1.760	2	2	2	−4	51.916	1.760	0.015
52.244	1.750	3	4	8	−2	52.242	1.750	−0.002
52.427	1.744	4	4	7	0	52.448	1.743	0.021
52.992	1.727	1	2	13	1	52.995	1.727	0.003
53.227	1.720	3	2	4	−4	53.242	1.719	0.015
53.676	1.706	5	3	10	1	53.685	1.706	0.009
54.003	1.697	3	1	16	0	53.966	1.698	−0.037
54.069	1.695	4	3	13	−1	54.061	1.695	−0.008
54.503	1.682	5	0	16	1	54.480	1.683	−0.023
54.730	1.676	3	3	3	−4	54.727	1.676	−0.003

Continued

https://doi.org/10.1017/S088571561500086X Published online by Cambridge University Press

TABLE I. Continued

$2\theta_{obs}$ (°)	d_{obs} (Å)	I_{obs}	h	k	l	$2\theta_{cal}$ (°)	d_{calc} (Å)	$\Delta 2\theta$
55.416	1.657	4	2	6	−4	55.402	1.657	−0.014
55.877	1.644	11	2	11	2	55.880	1.644	0.003
56.801	1.620	5	0	15	2	56.807	1.619	0.006
56.989	1.615	5	3	11	−3	56.990	1.615	0.000
57.397	1.604	2	1	14	2	57.405	1.604	0.008
57.589	1.599	2	3	6	−4	57.579	1.599	−0.010
57.985	1.589	2	0	17	−1	57.964	1.590	−0.022
58.161	1.585	4	1	17	−1	58.166	1.585	0.005
58.317	1.581	4	5	5	−1	58.305	1.581	−0.012
59.083	1.562	3	1	16	−2	59.088	1.562	0.005
59.432	1.554	3	5	6	−1	59.432	1.554	0.001
59.924	1.542	5	5	3	−3	59.923	1.542	−0.001
60.057	1.539	4	4	11	0	60.051	1.539	−0.006
60.384	1.532	2	1	15	2	60.377	1.532	−0.007
60.650	1.526	2	3	9	2	60.650	1.526	0.000
60.812	1.522	3	2	13	2	60.828	1.522	0.016
60.992	1.518	4	2	17	−1	60.985	1.518	−0.007
61.184	1.514	3	4	5	−4	61.193	1.513	0.010
61.455	1.508	3	3	15	0	61.445	1.508	−0.009
61.545	1.506	3	5	5	−3	61.532	1.506	−0.013
61.995	1.496	2	2	10	−4	61.978	1.496	−0.017
62.255	1.490	2	4	13	−1	62.249	1.490	−0.006
62.411	1.487	4	1	17	−2	62.407	1.487	−0.004
63.300	1.468	2	0	17	−2	63.291	1.468	−0.009
63.524	1.463	3	2	14	2	63.507	1.464	−0.017
64.053	1.453	3	2	17	−2	64.056	1.452	0.003
64.647	1.441	3	1	13	3	64.639	1.441	−0.008
64.839	1.437	4	2	18	0	64.850	1.437	0.011
65.346	1.427	3	1	19	−1	65.341	1.427	−0.005
65.797	1.418	3	4	14	−2	65.783	1.418	−0.014
66.302	1.409	3	2	15	2	66.317	1.408	0.015
66.688	1.401	4	5	3	−4	66.684	1.401	−0.004
67.195	1.392	3	4	6	2	67.197	1.392	0.002
67.465	1.387	3	1	8	4	67.457	1.387	−0.008
67.667	1.383	2	3	17	0	67.667	1.383	−0.001
67.981	1.378	3	2	19	−1	67.989	1.378	0.008
68.438	1.370	2	4	7	2	68.429	1.370	−0.010
68.620	1.367	3	2	18	1	68.621	1.367	0.001
68.873	1.362	3	0	20	−1	68.870	1.362	−0.003
69.041	1.359	3	1	20	−1	69.054	1.359	0.013
69.292	1.355	4	2	1	4	69.281	1.355	−0.011
69.955	1.344	3	1	18	2	69.959	1.344	0.004

space group $P2_1/n$ and unit-cell parameters: $a = 8.324(1)$ Å, $b = 27.838(2)$ Å, $c = 7.113(1)$, $\beta = 111.25(1)$, unit-cell volume $V = 1536.26$ Å^3, and $Z = 4$. The figures of merits are $F_{20} = 132.4(0.0044, 34)$ (Smith and Snyder, 1979) and $M_{20} = 48.4$ (de Wolf, 1968). All lines were indexed and are consistent with the $P2_1/n$ space group.

SUPPLEMENTARY MATERIALS AND METHODS

To view supplementary material for this article, Please visit http://dx.doi.org/10.1017/S088571561500086

ACKNOWLEDGMENT

This work was supported by the "Operational Program Prague – Competitiveness" (CZ.2.16/3.1.00/22197) and the National Programme of Sustainability (NPU I MSMT – LO1215 and NPU I MSMT – LO1304).

Boultif, A. and Louër, D. (**2004**). "Powder pattern indexing with the dichotomy method," J. Appl. Crystallogr. **37**, 724–731.

De Wolff, P. M. (**1968**). "A simplified criterion for the reliability of a powder pattern indexing," J. Appl. Crystallogr. **1**, 108–113.

Galanski, M., Arion, V. B., Jakupec, M. A., and Keppler, B. K. (**2003**). "Recent developments in the field of tumor-inhibiting metal complexes," Curr. Pharm. Des. **9**, 2078–2089.

Matthew, D. H. and Trevor, W. H. (**2002**). "Platinum(IV) antitumour compounds: their bioinorganic chemistry," Coordin. Chem. Rev.. **232**, 49–67.

Smith, G. S. and Snyder, R. L. (**1979**). "F_N: A criterion for rating powder diffraction patterns and evaluating the reliability of powder indexing," J. Appl. Crystallogr. **12**, 60–65.

Žák, F., Mistr, A., Poulová, A., Melka, M., Turánek, J., and Záluská, D. (**1999**). "Platinum complexes, its preparation and therapeutic application," World patent WO 9961451A1.