X-ray powder diffraction data for a DACH Pt iodide, *cis*-[diiodo(1*R*,2*R*)-1,2-diaminocyclohexane-$\kappa N, \kappa N'$] platinum(II)

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X-ray powder diffraction data, unit-cell parameters, and space group for a novel platinum-based anticancer complex *cis*-[diiodo(1*R*,2*R*)-1,2-diaminocyclo-hexane-$\kappa N, \kappa N'$] platinum(II), $Pt(C_6H_{14}N_2)I_2$, are presented [a=14.048(4) Å, b=7.588(3) Å, c=11.502(4) Å, β=98.446(5)°, space group $C2$, cell volume=1212.80 Å^3, and Z=4]. All measured lines were indexed and are consistent with the $C2$ space group. No detectable impurities were observed. © *2010 International Centre for Diffraction Data.* [DOI: 10.1154/1.3504593]

Key words: X-ray powder diffraction, crystal structure, DACH Pt iodide

I. INTRODUCTION

cis-[diiodo(1*R*,2*R*)-1,2-diaminocyclohexane (DACH)-$\kappa N, \kappa N'$] platinum(II) [$Pt(C_6H_{14}N_2)I_2$] is a new platinum-based anticancer complex. The search for novel platinum-based anticancer complexes remains an expanding area of the contemporary pharmaceutical industry. The driving force for investigations of this group of active anticancer therapeutics is the discovery of more active and less toxic analogues of the chemotherapy complexes used in today's clinical practice such as cisplatin, carboplatin, oxaliplatin, etc. (Ho *et al.*, 2003; Galanski *et al.*, 2003; Abu-Surrah and Kettunen, 2006). Some of the novel structures are based on the platinum (1*R*,2*R*)-1,2-DACH carrier ligand and various leaving groups bound to the central Pt metal atom.

II. SAMPLE PREPARATION

The title compound (Figure 1) was prepared from a solution of $K_2[PtCl_4]$ (0.029 mol, 12.0 g) in water (88 ml) mixed with the solution of KI (0.202 mol, 33.6 g) in water (29 ml). The mixture was stirred for 30 min and then a solution of DACH tartrate (0.032 mol, 8.51 g) in water (20 ml) was added. The *p*H value of the reaction mixture was adjusted with potassium hydroxide. The reaction mixture was stirred in the absence of light at 45 °C for 8 h. Subsequently, the product was filtered off from the suspension by filtration through sintered glass and dried in a vacuum drier. The resulting yellow powder, crude *cis*-[$Pt(DACH)I_2$], was obtained yielding 98.5%.

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 $K\alpha$ radiation (λ=1.5418 Å, generator setting: 40 kV, 30 mA). An ultrafast X'Celerator detector was employed to collect XRD data over the angular range from 7° to 70° 2θ with a step size of 0.017° 2θ and a counting time of 81.28 s/step. The experimental powder diffraction pattern is depicted in Figure 2 indicating that the powder specimen was preferred oriented. Data evaluation was performed using the software package HIGHSCORE PLUS V 2.2e PANalytical, Almelo, The Netherlands. The data collected are consistent with the monoclinic cell parameters [a=14.048(4) Å, b=7.588(3) Å, c=11.502(4) Å, β=98.446(5)°, space group $C2$ with cell volume=1212.80 Å^3, and Z=4]. These parameters were derived using DICVOL04 (Boultif and Louër, 2004) with the results all being within the errors indicated. The indexing results are listed in Table I. The following figures of merits were achieved: F_{20}=36.4 (0.0087, 63) (Smith and Snyder, 1979) and M_{20}=19.7.

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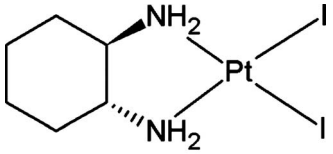

(I)

Figure 1. Structural formula of *cis*-DACH Pt iodide.

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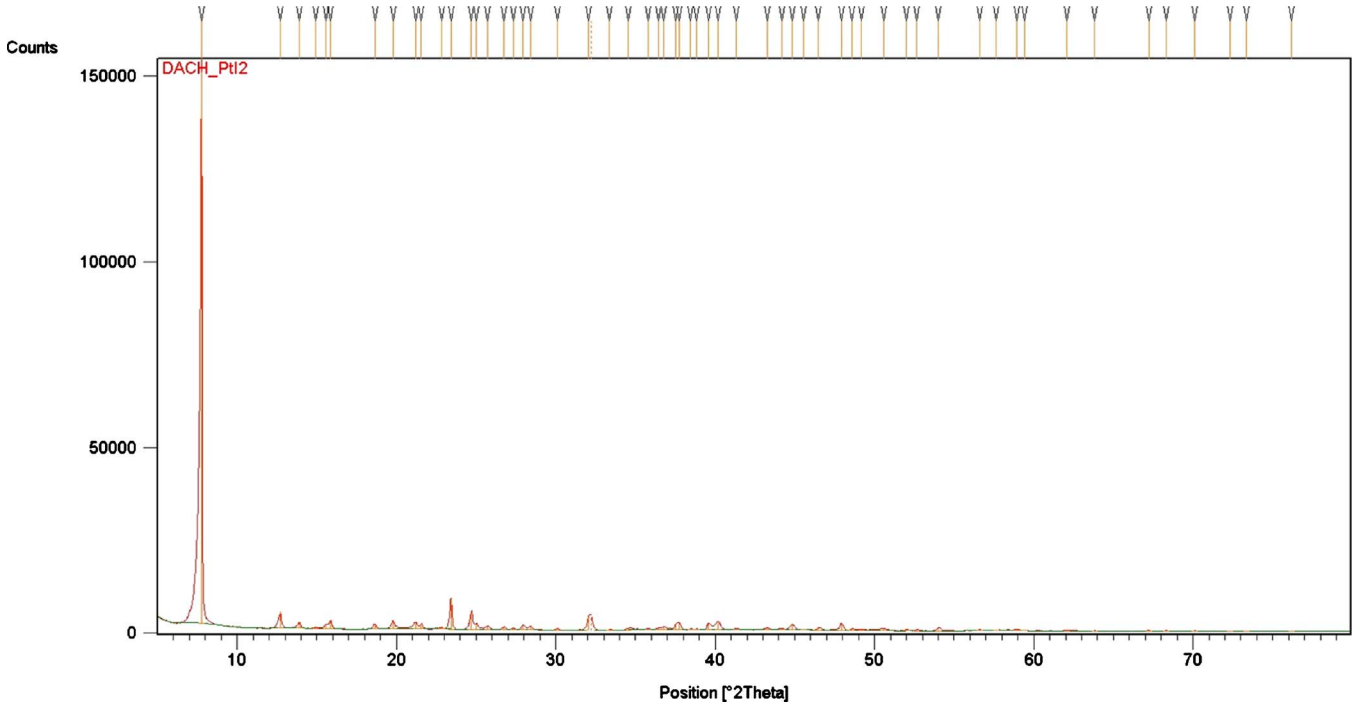

Figure 2. (Color online) X-ray powder diffraction pattern of *cis*-DACH Pt iodide using Cu $K\alpha$ radiation ($\lambda = 1.5418$ Å).

TABLE I. Indexed X-ray powder diffraction data for *cis*-DACH Pt iodide. Only the peaks with I_{rel} of 1 or greater are presented [$a = 14.048(4)$ Å, $b = 7.588(3)$ Å, $c = 11.502(4)$ Å, $\beta = 98.446(5)°$, $V = 1212.80$ Å^3, $Z = 4$, and space group $C2$]. All lines were indexed and are consistent with the $C2$ space group.

$2\theta_{obs}$ (deg)	d_{obs} (Å)	I_{obs}	h	k	l	$2\theta_{calc}$ (deg)	d_{calc} (Å)	$\Delta 2\theta$
7.756	11.390	100	0	0	1	7.764	11.378	−0.0119
12.706	6.962	3	2	0	0	12.731	6.948	−0.0136
13.892	6.369	1	2	0	−1	13.914	6.360	−0.0097
15.564	5.689	1	0	0	2	15.564	5.689	0.0000
15.840	5.590	1	1	1	1	15.873	5.579	−0.0116
18.623	4.761	1	2	0	−2	18.636	4.757	−0.0034
19.790	4.483	1	1	1	−2	19.783	4.484	0.0016
21.187	4.190	1	1	1	2	21.226	4.183	−0.0075
21.555	4.119	1	2	0	2	21.577	4.115	−0.0041
23.424	3.795	6	0	2	0	23.429	3.794	−0.0008
24.703	3.601	3	0	2	−1	24.717	3.599	−0.0020
25.008	3.558	1	2	0	−3	25.023	3.556	−0.0020
25.709	3.462	1	4	0	−1	25.668	3.468	0.0055
27.954	3.189	1	4	0	1	27.911	3.194	0.0049
28.371	3.143	1	2	0	3	28.396	3.141	−0.0027
32.156	2.781	2	2	0	−4	32.180	2.779	−0.0020
36.768	2.442	1	4	2	1	36.752	2.443	0.0010
37.599	2.390	1	4	0	3	37.561	2.393	0.0023
37.741	2.382	1	4	0	−4	37.789	2.379	−0.0029
39.579	2.275	1	0	0	5	39.573	2.276	0.0003
40.177	2.243	1	2	2	−4	40.188	2.242	−0.0006
44.818	2.021	1	3	3	2	44.824	2.020	−0.0002
46.539	1.950	1	0	2	−5	46.499	1.951	0.0016
47.929	1.896	1	0	0	6	47.935	1.896	−0.0002
54.011	1.696	1	0	4	−3	54.006	1.697	0.0002

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