X-ray powder diffraction data for methoxetamine hydrochloride, $C_{15}H_{22}ClNO_2$

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X-ray powder diffraction data, unit-cell parameters and space group for 2-(ethylamino)-2-(3-methoxyphenyl)cyclohexan-1-one hydrochloride, $C_{15}H_{22}ClNO_2$, are reported [$a = 8.574(2)$ Å, $b = 9.943(2)$ Å, $c = 8.774(1)$ Å, $\beta = 100.294(3)°$, unit-cell volume $V = 736(1)$ Å^3, $Z = 2$, and space-group $P2_1$]. All measured lines were indexed and are consistent with the $P2_1$ space group. No detectable impurities were observed. © *2017 International Centre for Diffraction Data.*
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Key words: X-ray powder diffraction, methoxetamine, NPS, new psychoactive substances, designer drugs, dissociative anesthetics

I. INTRODUCTION

Methoxetamine, MXE [2-(ethylamino)-2-(3-methoxyphenyl)cyclohexan-1-one] is a structural and pharmacological analog of ketamine (Morris and Wallach, 2014). For the first time it was synthetized in the UK by a synthetic chemist known as "M". Structurally MXE belongs to the group of arylcyclohexylamines and has anesthetic and sedative effects (Zanda *et al.*, 2016). This designer drug emerged on the black market in 2010 and by 2015 was involved in 120 non-fatal and 22 fatal intoxications (EMCDDA, 2014; Zanda *et al.*, 2016). Though important pharmacological data have been published (e.g. metabolism, behavioral tests), there is still the lack of information about this compound (Menzies *et al.*, 2014; Zawilska, 2014; Hajkova *et al.*, 2016; Horsley *et al.*, 2016; Zanda *et al.*, 2016). MXE had been offered as the legal alternative for ketamine, but because of the dozens of severe intoxications it was banned in most EU states (Jurasek and Kuchar, 2016; Zanda *et al.*, 2016). Even though MXE (Figure 1) is banned by law, its analogs are available on the black market and may be still legal (e.g. methoxphenidine and deschloroketamine) (Hofer *et al.*, 2014; Frison *et al.*, 2016).

We have not found this compound in the CSD database or in the PDF4+ database (Allen, 2002; ICDD, 2015). Therefore, we have decided to characterize this compound by X-ray powder diffraction (XRD) technique. In our study, we present powder data for MXE hydrochloride ($C_{15}H_{22}ClNO_2$).

II. EXPERIMENTAL

A. Synthesis

The synthesis of MXE hydrochloride was carried out according to Hays *et al.* (2012) instructions and Stevens and Parke (1966) patent. 1-((Ethylimino)(3-methoxyphenyl) methyl)cyclopentan-1-ol (600 mg, 2.4 mmol) was dissolved in decalin (2 ml) and stirred 15 h at 190 °C in a microwave reactor. The reaction mixture was cooled, diluted with 15% hydrochloric acid, and extracted with dichloromethane. The aqueous layer was separated, and then it was made alkaline and extracted with diethyl ether. The organic layer was dried over $MgSO_4$ and suction filtered. The filtrate was treated with a solution of hydrogen chloride in diethyl ether and the solvent was evaporated. Further purification was done by recrystallization from isopropyl alcohol. MXE hydrochloride was isolated as a yellowish solid and confirmed by NMR (nuclear magnetic resonance) analysis (130 mg, 19% yield) (Stevens and Parke, 1966; Hays *et al.*, 2012).

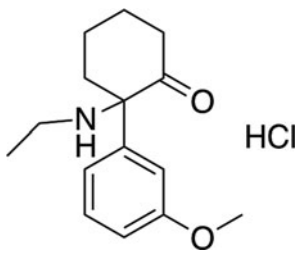

Figure 1. Structural formula of 2-(ethylamino)-2-(3-methoxyphenyl) cyclohexan-1-one hydrochloride.

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TABLE I. Indexed X-ray powder diffraction data for $C_{15}H_{22}ClNO_2$.

$2\theta_{obs}$ (°)	d_{obs} (Å)	I_{obs}	H	k	l	$2\theta_{cal}$ (°)	d_{calc} (Å)	$\Delta 2\theta$
10.270	8.6064	52	0	0	1	10.238	8.6332	−0.032
13.325	6.6392	13	1	0	−1	13.289	6.6574	−0.037
13.600	6.5057	62	0	1	−1	13.573	6.5188	−0.027
13.778	6.4223	24	1	1	0	13.756	6.4324	−0.022
15.955	5.5503	51	1	0	1	15.935	5.5574	−0.021
17.855	4.9638	3	0	2	0	17.828	4.9714	−0.028
18.290	4.8467	29	1	1	1	18.273	4.8511	−0.017
20.592	4.3097	69	0	2	1	20.600	4.3081	0.008
21.394	4.1500	14	1	0	−2	21.365	4.1556	−0.029
21.730	4.0866	10	2	0	−1	21.718	4.0888	−0.012
22.410	3.9641	2	0	1	−2	22.436	3.9595	0.026
22.917	3.8775	10	2	1	0	22.885	3.8828	−0.032
23.207	3.8297	100	1	1	−2	23.179	3.8342	−0.028
23.520	3.7795	70	2	1	−1	23.507	3.7815	−0.013
24.013	3.7030	39	1	2	1	23.998	3.7052	−0.015
24.794	3.5881	3	1	0	2	24.772	3.5912	−0.022
26.388	3.3749	54	1	1	2	26.365	3.3777	−0.022
26.665	3.3404	9	2	1	1	26.656	3.3415	−0.009
27.360	3.2571	19	0	2	2	27.341	3.2594	−0.019
27.726	3.2149	8	2	2	0	27.715	3.2162	−0.012
27.979	3.1864	8	1	2	−2	27.961	3.1884	−0.018
28.268	3.1545	2	2	1	−2	28.250	3.1565	−0.018
28.867	3.0904	14	0	3	1	28.832	3.0941	−0.035
30.109	2.9657	5	1	3	−1	30.096	2.9669	−0.013
30.712	2.9089	5	1	2	2	30.687	2.9111	−0.024
30.977	2.8845	9	1	0	−3	30.964	2.8857	−0.013
32.357	2.7646	16	0	1	3	32.361	2.7643	0.004
32.908	2.7196	9	3	1	−1	32.918	2.7188	0.010
33.038	2.7091	8	3	1	0	33.081	2.7057	0.043
33.464	2.6756	7	2	1	2	33.457	2.6762	−0.007
34.090	2.6279	2	0	3	2	34.078	2.6288	−0.011
34.423	2.6032	4	2	0	−3	34.418	2.6036	−0.005
34.710	2.5824	15	1	0	3	34.659	2.5860	−0.051
35.255	2.5437	4	3	0	1	35.263	2.5431	0.008
35.628	2.5179	3	2	1	−3	35.617	2.5187	−0.011
35.978	2.4942	27	3	1	−2	35.987	2.4936	0.009
36.451	2.4630	7	3	1	1	36.438	2.4638	−0.013
37.010	2.4270	1	2	2	2	37.033	2.4255	0.023
37.643	2.3876	3	0	4	−1	37.626	2.3886	−0.017
38.300	2.3482	1	2	3	−2	38.292	2.3486	−0.007
38.633	2.3287	2	1	4	−1	38.634	2.3287	0.001
39.257	2.2931	4	1	2	3	39.237	2.2942	−0.020
39.759	2.2653	2	3	2	1	39.781	2.2641	0.023
40.641	2.2181	2	3	0	−3	40.622	2.2191	−0.019
40.976	2.2008	13	2	0	3	40.971	2.2011	−0.005
41.541	2.1722	4	0	3	−3	41.526	2.1729	−0.015
41.981	2.1504	10	3	3	−1	41.977	2.1506	−0.004
42.320	2.1340	4	3	1	2	42.331	2.1334	0.011
42.852	2.1087	2	4	0	0	42.848	2.1089	−0.004
43.525	2.0776	3	2	0	−4	43.521	2.0778	−0.005
43.821	2.0643	2	4	1	0	43.850	2.0630	0.028
44.217	2.0467	4	2	3	−3	44.201	2.0474	−0.015
44.514	2.0337	3	3	3	−2	44.511	2.0339	−0.003
44.840	2.0197	2	3	3	1	44.889	2.0176	0.049
45.295	2.0005	6	3	2	2	45.311	1.9998	0.017
45.791	1.9800	7	0	2	4	45.795	1.9798	0.005
46.118	1.9667	2	1	1	4	46.111	1.9669	−0.007
46.763	1.9410	1	4	2	0	46.753	1.9414	−0.011
46.902	1.9356	1	1	5	0	46.905	1.9355	0.002
48.031	1.8927	2	4	2	−2	48.083	1.8908	0.052
48.254	1.8845	4	1	4	−3	48.285	1.8833	0.032
48.911	1.8607	2	1	2	4	48.910	1.8607	−0.002
49.210	1.8501	4	3	1	−4	49.229	1.8494	0.019
49.999	1.8227	6	3	3	2	49.967	1.8238	−0.032
50.791	1.7962	2	2	0	4	50.807	1.7956	0.016
51.324	1.7787	1	4	3	0	51.309	1.7792	−0.015
51.923	1.7596	1	3	2	−4	51.901	1.7603	−0.022
52.100	1.7540	2	1	0	−5	52.093	1.7543	−0.007
52.308	1.7476	4	4	2	−3	52.285	1.7483	−0.023
52.986	1.7268	2	0	0	5	52.991	1.7266	0.005
53.309	1.7171	2	1	3	4	53.328	1.7165	0.019
53.874	1.7004	1	0	1	−5	53.848	1.7012	−0.027
54.373	1.6860	1	5	0	0	54.333	1.6871	−0.040
55.491	1.6546	2	1	2	−5	55.503	1.6543	0.012
56.188	1.6357	2	0	5	3	56.180	1.6360	−0.008
56.501	1.6274	1	0	6	−1	56.501	1.6274	0.000
58.379	1.5795	1	4	4	−2	58.399	1.5790	0.021
59.096	1.5620	1	3	1	4	59.119	1.5614	0.024
59.453	1.5535	1	1	2	5	59.461	1.5533	0.008
60.954	1.5188	1	2	3	−5	60.959	1.5186	0.006
61.517	1.5062	1	2	0	5	61.511	1.5063	−0.006
62.501	1.4848	1	3	4	3	62.476	1.4854	−0.025
62.924	1.4759	1	2	5	3	62.939	1.4755	0.015
63.879	1.4561	1	4	5	−1	63.869	1.4563	−0.011
64.442	1.4447	1	3	3	−5	64.458	1.4444	0.016
65.011	1.4334	1	1	4	−5	65.019	1.4333	0.009

Only the peaks with I_{rel} of 1 or greater are presented [$a = 8.574(2)$ Å, $b = 9.943(2)$ Å, $c = 8.774(1)$ Å, $\beta = 100.294(3)°$, unit-cell volume $V = 736(1)$ Å^3, $Z = 2$, and space-group $P2_1$]. All lines were indexed and are consistent with the $P2_1$ space group. The d-values were calculated using Cu$K\alpha_1$ radiation ($\lambda = 1.5406$ Å).

B. Specimen preparation

The sample was prepared by careful grinding in an agate mortar and was then front-loaded into the specimen holder.

C. Diffraction data collection and reduction

The diffraction pattern for the title compound was collected at room temperature with an X'Pert^3 Powder θ–θ powder diffractometer with parafocusing Bragg–Brentano geometry using Cu$K\alpha$ radiation ($\lambda = 1.5418$ Å, Ni filter, generator setting: 40 kV, 30 mA). An ultrafast PIXCEL detector was employed to collect XRD data over the angular range from 5 to 70° 2θ with a step size of 0.013° 2θ and a counting time of 316.97 s step^{-1}.

The software package HighScore Plus V 3.0e (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 the smoothed peaks were used to determine the peak positions and intensities of the diffraction peaks (Table 1). The d-values were calculated using Cu$K\alpha_1$ radiation ($\lambda = 1.5406$ Å).

III. RESULTS AND DISCUSSION

The automatic indexing of results was obtained using TREOR (Werner *et al.*, 1985). The experimental powder diffraction pattern is showed in Figure 2. MXE hydrochloride, $C_{15}H_{22}ClNO_2$, is monoclinic with space group $P2_1$ and unit-cell parameters: $a = 8.574(2)$ Å, $b = 9.943(2)$ Å, $c = 8.774(1)$ Å, $\beta = 100.294(3)°$, unit-cell volume $V = 736(1)$ Å^3, and $Z = 2$. The figures of merits are $F_{20} = 45(0.0161, 28)$ and $M_{20} = 22$ (de Wolff, 1968; Smith and Snyder, 1979). All measured lines (Table 1) were indexed and are consistent with the $P2_1$ space group.

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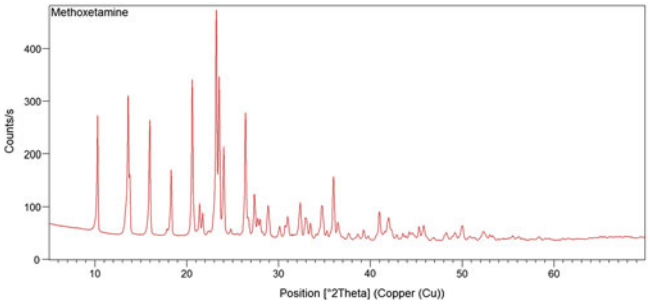

Figure 2. X-ray powder diffraction pattern of title compound using Cu$K\alpha$ radiation ($\lambda = 1.5418$ Å).

The single-crystal experiment was done at the temperature of 190 K and the structure solution was obtained. The title compound is monoclinic with space group $P2_1$ and unit-cell parameters: $a = 8.5360(7)$ Å, $b = 9.9155(9)$ Å, $c = 8.7558(8)$ Å, $\beta = 100.354(2)°$, unit-cell volume $V = 729.0(7)$ Å^3, and $Z = 2$. The difference in unit-cell parameters from the single-crystal data and the powder diffraction data is because of the temperature expansion.

SUPPLEMENTARY MATERIAL

The supplementary material for this article can be found at https://doi.org/10.1017/S0885715617000860.

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