X-ray powder diffraction data for methoxmetamine hydrochloride, $C_{14}H_{20}ClNO_2$

Bronislav Jurasek,[1,2] Martin Babor,[3] Stepan Huber,[4] and Martin Kuchar[1,2,a)]
[1]*Department of Chemistry of Natural Compounds UCT Prague, Technická 5, Prague, 166 28, Czech Republic*
[2]*Forensic Laboratory of Biologically Active Substances UCT Prague, Technická 5, Prague, 166 28, Czech Republic*
[3]*Department of Solid State Chemistry UCT Prague, Technická 5, Prague, 166 28, Czech Republic*
[4]*Department of Inorganic Chemistry UCT Prague, Technická 5, Prague, 166 28, Czech Republic*

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X-ray powder diffraction data, unit-cell parameters and space group for methoxmetamine ($C_{14}H_{20}ClNO_2$) are reported [$a = 6.5768(7)$ Å, $b = 14.0830(10)$ Å, $c = 15.0530(10)$ Å, $\beta = 90.975(2)°$, unit-cell volume $V = 1394.0(2)$ Å^3, and $Z = 4$, and space group $P2_1/n$]. All measured lines were indexed and are consistent with the $P2_1/n$ space group. No detectable impurities were observed.
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Key words: new psychoactive substances, X-ray powder diffraction, methoxmetamine, designer drugs, dissociative anesthetics

I. INTRODUCTION

New psychoactive substances (NPS) are pharmacologically unidentified xenobiotics with unknown effects and toxicity that mimic the effects of already forbidden psychoactive substances by modification of their chemical structure while maintaining the pharmacophore. There is a significant increase in the prevalence of NPS within the population which is likely to be caused by availability over the Internet and their temporary legality. The popularity of these substances has increased significantly over the last few years. While more than 560 NPS were monitored in the European market in 2015, the total number of monitored substances increased to more than 620 in 2016 (Hondebrink *et al*., 2015; EMCDDA, 2016; EMCDDA and Europol, 2017). The legislative regulation is complicated mainly because of the large increase of new substances occurring every year and consequent availability through Internet sales (Fojtikova *et al*., 2017). These compounds are associated with considerable health risks (including fatal intoxications) for the consumers (Páleníček *et al*., 2016; Jurasek and Kuchar, 2017). European Monitoring Centre for Drugs and Drug Addiction (EMCDDA) distinguishes several groups of NPS (e.g. synthetic cannabinoids, cathinones, and dissociative anesthetics).

Methoxmetamine (MMXE, Figure 1, structure 1) is an arylcyclohexylamine that belongs to the group of dissociative anesthetics. It is an analog of methoxetamine (MXE, Figure 1, structure 2) which was associated with 120 non-fatal intoxications and 22 deaths (reported by EMCDDA Early Warning System) (EMCDDA, 2014; Zanda *et al*., 2016; Jurásek *et al*., 2017).

II. EXPERIMENTAL

A. Synthesis

The synthesis of MMXE (Figure 1, structure 1) hydrochloride was carried out according to Stevens and Parke (1966) patent and Jurasek *et al*. (2017) instructions. 1-((Methylimino)(3-methoxyphenyl)methyl)cyclopentan-1-ol (6.4 g, 27.4 mmol) was dissolved in decalin (12 ml) and it was stirred for 4 h at 190 °C in a microwave reactor. The reaction mixture was cooled and treated with a solution of hydrogen chloride in diethyl ether. The precipitated solid was suction filtered, washed with acetone, and recrystallized from a methanol/diethyl ether mixture. MMXE (1) hydrochloride was isolated as a yellowish solid and confirmed by NMR analysis (1.5 g, 20% yield) (Stevens and Parke, 1966; Jurasek *et al*., 2017).

B. Specimen preparation

The grinded sample was loaded into the specimen holder.

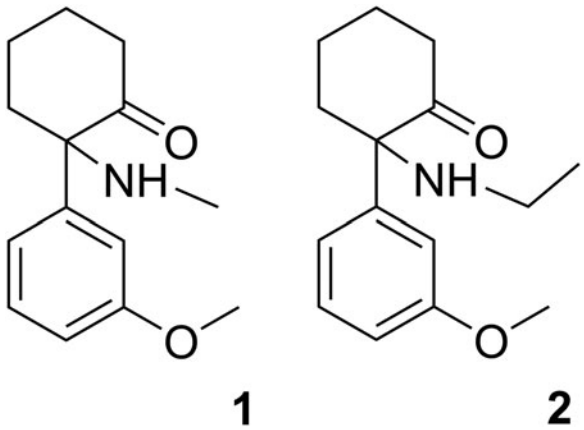

Figure 1. MMXE (1) and MXE (2).

a)Author to whom correspondence should be addressed. Electronic mail: kuchara@vscht.cz

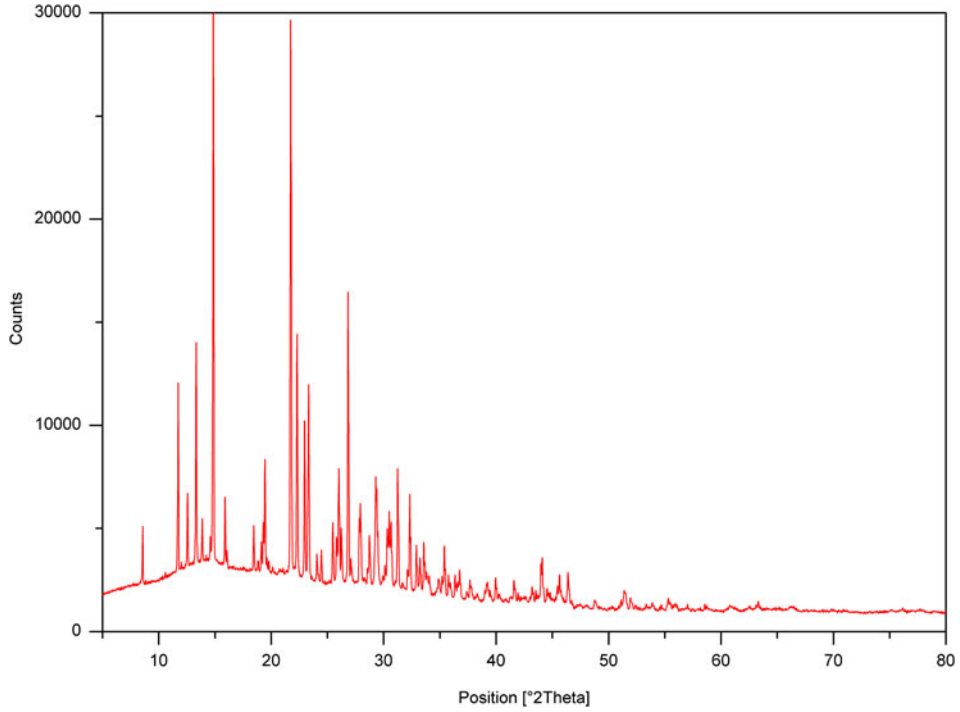

Figure 2. (Color online) X-ray powder diffraction pattern of MMXE hydrochloride using Cu$K\alpha$ radiation (λ = 1.5418 Å). See more detail in the supplementary material.

TABLE I. Indexed X-ray powder diffraction data for $C_{14}H_{20}ClNO_2$. Only the peaks with the I_{rel} of 1 or greater are presented [a = 6.5768(7) Å, b = 14.0830(10) Å, c = 15.0530(10) Å, β = 90.975(2)°, unit-cell volume V = 1394.0(2) 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 (λ = 1.5406 Å).

$2\theta_{obs}$ (deg)	d_{obs} (Å)	I_{obs}	h	k	l	$2\theta_{calc}$ (deg)	d_{calc} (Å)	$\Delta 2\theta$ (deg)
8.570	10.3098	8	0	1	1	8.592	10.2831	0.022
11.731	7.5375	28	0	0	2	11.751	7.5252	0.019
12.546	7.0501	11	0	2	0	12.561	7.0413	0.016
13.310	6.6467	34	0	1	2	13.330	6.6371	0.019
13.855	6.3866	7	0	2	1	13.874	6.3779	0.019
14.576	6.0721	4	1	0	−1	14.596	6.0637	0.020
14.839	5.9651	96[a]	1	1	0	14.856	5.9583	0.017
15.881	5.5761	11	1	1	−1	15.900	5.5694	0.019
16.050	5.5178	2	1	1	1	16.070	5.5109	0.020
18.436	4.8086	8	1	2	0	18.446	4.8060	0.010
18.825	4.7101	2	1	1	−2	18.839	4.7068	0.013
19.115	4.6393	5	1	1	2	19.126	4.6366	0.011
19.286	4.5985	8	1	2	−1	19.302	4.5948	0.016
19.430	4.5647	19	1	2	1	19.443	4.5618	0.013
19.781	4.4846	2	0	3	1	19.796	4.4813	0.015
21.724	4.0877	100[a]	0	2	3	21.734	4.0858	0.010
22.290	3.9852	42	0	3	2	22.303	3.9828	0.013
22.964	3.8696	30	1	1	−3	22.979	3.8671	0.015
23.317	3.8119	30[a]	1	1	3	23.336	3.8088	0.019
24.058	3.6961	5	1	3	1	24.069	3.6945	0.011
24.452	3.6375	5	0	1	4	24.468	3.6351	0.016
25.474	3.4938	11	1	2	−3	25.486	3.4922	0.012
25.802	3.4501	9	1	2	3	25.810	3.4491	0.007
26.007	3.4234	17[a]	1	3	−2	26.030	3.4204	0.024
26.234	3.3943	10	1	3	2	26.243	3.3932	0.009
26.831	3.3201	55	0	2	4	26.844	3.3185	0.013
27.089	3.2891	4	2	0	0	27.099	3.2879	0.010
27.835	3.2026	12[a]	2	1	0	27.842	3.2018	0.007
27.943	3.1904	12	0	4	2	27.957	3.1889	0.014
28.307	3.1502	1	2	1	−1	28.380	3.1423	0.073
28.570	3.1218	3	2	1	1	28.576	3.1212	0.006

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TABLE I. Continued

$2\theta_{obs}$ (deg)	d_{obs} (Å)	I_{obs}	h	k	l	$2\theta_{calc}$ (deg)	d_{calc} (Å)	$\Delta 2\theta$ (deg)
28.733	3.1045	9	1	4	0	28.740	3.1038	0.006
29.300	3.0457	18[a]	1	4	−1	29.310	3.0447	0.010
29.400	3.0356	12[a]	1	4	1	29.405	3.0350	0.005
29.498	3.0257	5	1	3	3	29.504	3.0252	0.005
29.949	2.9812	2[a]	1	2	−4	29.954	2.9807	0.005
30.122	2.9644	4	2	1	−2	30.127	2.9640	0.005
30.322	2.9454	10[a]	1	2	4	30.326	2.9449	0.005
30.492	2.9293	12[a]	2	1	2	30.498	2.9288	0.005
30.650	2.9145	12	2	2	1	30.657	2.9139	0.007
31.233	2.8615	23	1	4	2	31.236	2.8612	0.003
32.106	2.7856	4	2	2	−2	32.117	2.7847	0.011
32.313	2.7683	18[a]	0	5	1	32.310	2.7685	−0.002
32.902	2.7200	9[a]	1	0	5	32.909	2.7195	0.006
33.224	2.6944	6[a]	1	3	−4	33.227	2.6942	0.002
33.563	2.6680	10[a]	1	3	4	33.567	2.6677	0.004
33.811	2.6490	3	1	4	−3	33.810	2.6490	−0.001
34.053	2.6307	3	1	4	3	34.061	2.6301	0.008
34.881	2.5701	3	0	4	4	34.872	2.5708	−0.010
35.180	2.5490	4	1	5	−1	35.183	2.5487	0.003
35.389	2.5344	10[a]	0	3	5	35.396	2.5339	0.007
35.759	2.5090	4	0	0	6	35.768	2.5084	0.009
35.940	2.4968	3	2	0	−4	35.937	2.4970	−0.003
36.342	2.4700	5	0	1	6	36.350	2.4695	0.008
36.560	2.4559	3[a]	0	5	3	36.558	2.4560	−0.002
36.753	2.4434	5	1	5	2	36.756	2.4432	0.004
37.385	2.4035	2[a]	1	4	−4	37.380	2.4038	−0.005
37.666	2.3863	4[a]	2	3	−3	37.659	2.3867	−0.007
37.822	2.3768	1	2	4	−1	37.809	2.3775	−0.013
38.252	2.3510	1[a]	1	3	5	38.216	2.3531	−0.036
39.004	2.3074	2	1	5	−3	39.010	2.3070	0.006
39.191	2.2968	3[a]	2	4	−2	39.181	2.2974	−0.010
39.476	2.2809	2	2	4	2	39.476	2.2809	−0.001
39.950	2.2549	5	0	5	4	39.952	2.2548	0.002
40.229	2.2399	1	0	6	2	40.215	2.2407	−0.014
41.259	2.1863	1	1	6	1	41.280	2.1853	0.021
41.589	2.1698	4	1	4	−5	41.593	2.1696	0.005
41.943	2.1522	2	1	4	5	41.944	2.1522	0.001
43.007	2.1015	1	2	2	5	43.010	2.1013	0.004
43.201	2.0925	3[a]	3	2	0	43.192	2.0929	−0.009
43.529	2.0774	2	3	2	−1	43.527	2.0775	−0.003
43.995	2.0565	8[a]	0	5	5	43.993	2.0566	−0.002
44.120	2.0510	5	2	5	2	44.107	2.0515	−0.013
44.538	2.0327	3[a]	1	1	−7	44.544	2.0324	0.006
44.802	2.0213	2[a]	3	0	−3	44.804	2.0212	0.002
45.460	1.9936	3[a]	2	3	5	45.468	1.9933	0.008
45.647	1.9858	6	3	3	0	45.641	1.9861	−0.006
45.872	1.9766	2[a]	3	1	3	45.878	1.9764	0.006
46.395	1.9556	6[a]	1	5	5	46.374	1.9564	−0.020
46.702	1.9434	1[a]	1	4	6	46.705	1.9433	0.002
47.486	1.9131	1	3	3	2	47.488	1.9131	0.002
48.765	1.8659	2[a]	1	3	7	48.766	1.8659	0.002
49.997	1.8228	1	2	3	6	49.994	1.8229	−0.003
51.098	1.7861	2[a]	1	1	8	51.097	1.7861	−0.002
51.388	1.7767	4	1	6	5	51.382	1.7769	−0.005
51.938	1.7591	3	1	2	−8	51.933	1.7593	−0.006
52.414	1.7443	1[a]	3	1	5	52.416	1.7442	0.002
53.355	1.7157	1[a]	3	5	1	53.349	1.7159	−0.006
53.885	1.7001	1	1	8	0	53.872	1.7005	−0.013
54.697	1.6767	1	2	7	−2	54.712	1.6763	0.014
55.298	1.6599	2	1	8	−2	55.288	1.6602	−0.010
55.528	1.6536	1	1	6	6	55.522	1.6538	−0.006
55.880	1.6440	1	4	0	0	55.883	1.6440	0.003
57.022	1.6138	1[a]	1	0	9	57.008	1.6141	−0.014
58.549	1.5753	1	0	3	9	58.548	1.5753	−0.001
60.803	1.5222	1[a]	1	9	0	60.800	1.5222	−0.004
62.499	1.4849	1[a]	2	7	5	62.486	1.4851	−0.013

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TABLE I. Continued

$2\theta_{obs}$ (deg)	d_{obs} (Å)	I_{obs}	h	k	l	$2\theta_{calc}$ (deg)	d_{calc} (Å)	$\Delta 2\theta$ (deg)
63.319	1.4676	2[a]	3	6	4	63.329	1.4674	0.011
66.127	1.4119	1	2	3	9	66.130	1.4119	0.003
69.791	1.3465	1	4	6	0	69.789	1.3465	−0.002
76.138	1.2493	1	0	1	12	76.137	1.2493	0.000

[a]Implies possible multiple line contributions.

C. Diffraction data collection and reduction

X-ray powder diffraction data were collected at room temperature on a Bruker D8 Discover (Bruker, Germany) powder diffractometer with a parafocusing Bragg–Brentano geometry using CuK_α radiation ($\lambda = 1.5418$ Å, $U = 40$ kV, $I = 40$ mA) (Figure 2). The ratio of the source was 10:5:2 (Cu$K\alpha_1$:$K\alpha_2$:K_β) and selected specific energy from the detector was used for filtering K_β. No K_β filter was used. Data were scanned over the angular range of 5–80° (2θ) with a step size of 0.02° (2θ).

HighScore Plus 3.0a (PANalytical, Almelo, Netherlands) software was used to fit the background using a polynomial method, to smooth the data and to eliminate the $K\alpha_2$ component. The top of the smoothed peaks was 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

Indexation was made by using the algorithm TREOR (Werner, 1964). MMXE (1) ($C_{14}H_{20}ClNO_2$) was crystallized in a monoclinic crystal system with the space group $P2_1/n$ and unit-cell parameters: $a = 6.5768(7)$ Å, $b = 14.0830(10)$ Å, $c = 15.0530(10)$ Å, $\beta = 90.975(2)°$, unit-cell volume $V = 1394.0(2)$ Å^3, and $Z = 4$. The figures of merits are $\underline{F}_{20} = 97$ (0.005474, 38) $M_{20} = 43$ (de Wolff, 1968; Smith and Snyder, 1979). All measured lines were indexed and are consistent with the $P2_1/n$ space group.

The primary cell was also confirmed by single crystal X-ray diffraction. The crystal was measured at 190 K, the structure was solved, and will be described in detail in a publication focused on MMXE and MXE structures. The compound crystallized in the monoclinic crystal system with the space group $P2_1/n$ and unit-cell parameters: $a = 6.5578(6)$ Å, $b = 13.8927(15)$ Å, $c = 15.006(3)$ Å, $\beta = 91.066(11)°$, unit-cell volume $V = 1366.9(3)$ Å^3, and $Z = 4$. The small differences between these primary cells are because there is temperature expansion.

SUPPLEMENTARY MATERIAL

The supplementary material for this article can be found at https://doi.org/10.1017/S0885715618000532.

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CONFLICTS OF INTEREST

The authors have no conflicts of interest to declare.

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