



X-ray powder diffraction data for three new compounds obtained as a result of CO<sub>2</sub> captureKlaudia Nowakowska <sup>1</sup> and Wiesław Łasocha <sup>1,2,a)</sup><sup>1</sup>Faculty of Chemistry, Jagiellonian University, Gronostajowa 2, 30-387 Krakow, Poland<sup>2</sup>Jerzy Haber Institute of Catalysis and Surface Chemistry PAS, Niezapominajek 8, 30-239 Krakow, Poland

(Received 25 May 2023; accepted 18 October 2023)

The field of research related to CO<sub>2</sub> capture is significant and really attractive for sustainable green chemistry. Focusing attention on this topic in our research led to obtaining new compounds based on diamines. As a result of the syntheses carried out using aqueous solutions of diamines exposed to the slow action of carbon dioxide from the air, three new monocarbamates were obtained. X-ray powder diffraction data for the obtained compounds: **12-propCO<sub>2</sub>** (C<sub>4</sub>H<sub>10</sub>N<sub>2</sub>O<sub>2</sub>) [ $a = 9.3033(7)$ ,  $b = 9.2485(7)$ ,  $c = 7.4735(7)$  Å,  $\beta = 111.214(7)^\circ$ ,  $V = 599.46$  Å<sup>3</sup>,  $Z = 4$ , space group *Ia*]; **13-propCO<sub>2</sub>** (C<sub>4</sub>H<sub>10</sub>N<sub>2</sub>O<sub>2</sub>) [ $a = 5.0065(10)$ ,  $b = 12.2093(23)$ ,  $c = 4.9006(10)$  Å,  $\beta = 96.457(18)^\circ$ ,  $V = 297.65$  Å<sup>3</sup>,  $Z = 2$ , space group *P2*<sub>1</sub>]; and **13-dytekCO<sub>2</sub>** (C<sub>6</sub>H<sub>14</sub>N<sub>2</sub>O<sub>2</sub>) [ $a = 28.374(3)$ ,  $c = 5.1726(9)$  Å,  $V = 3606.53$  Å<sup>3</sup>,  $Z = 18$ , space group *R*<sup>3</sup>̄] are reported in this paper.

© The Author(s), 2023. Published by Cambridge University Press on behalf of International Centre for Diffraction Data.

[doi:10.1017/S0885715623000404]

Keywords: carbamate, CO<sub>2</sub> capture, aliphatic amines, X-ray diffraction

## I. INTRODUCTION

Amines have the ability to bond with carbon dioxide gas, which leads to the formation of compounds called carbonates or carbamates (Zhou et al., 2007; Leszczyński et al., 2022). Aqueous solutions of selected aliphatic diamines were exposed to the slow action of carbon dioxide from air under normal conditions. As a result of the syntheses, several products were obtained and were investigated by performing chemical analyses and recording diffraction patterns.

Studies devoted to these compounds seem to be significant in the analysis of products formed on various types of surfaces near the plugs and valves of tanks containing amines or their solutions. In the case of the synthesis of chemical compounds with the participation of amines in aqueous solutions, there is a high probability of contamination of these compounds by products similar to those presented in this work. These impurities may be formed in parallel with the main product, or the obtained main product may slowly decompose under the influence of the aggressive action of carbon dioxide. Furthermore, understanding the reactivity and nature of the formation of the studied compounds may be useful from the point of view of green chemistry and in the broad sense of research in the field of “carbon dioxide capture”, which will undoubtedly contribute to environmental protection.

Products formed in the reaction of aliphatic diamines with metal halides [for instance, coordination polymers  $\text{MX}_2 \leftarrow \text{NH}_2 - (\text{CH}_2)_n - \text{NH}_2 \rightarrow \text{MX}_2$ , hybrid compounds ZnS-1,3-diaminopropane (Luberda-Durnaś et al., 2011; Gonzalez et al., 2019)], stored under normal conditions and dried in the air, exhibit higher carbon contents than expected from

the structural data. Our research may contribute to explaining the causes of this frequently observed phenomenon.

In fact, powder diffraction data for compounds denoted as **12-propCO<sub>2</sub>** and **13-dytekCO<sub>2</sub>** corrects diffraction data submitted by us previously to the PDF-4+ database (Gates-Rector et al., 2019). Trying to obtain new molybdates of 1,2-diaminopropane and 1,3-diaminopentane, MoO<sub>3</sub> was dissolved in excess of the relevant amine and water. By slow evaporation in air white precipitates (usually wet and greasy) were obtained. Diffraction patterns for the obtained solids were indexed, and deposited as entries 00-61-1246 for 12-prop and 00-62-0980 in the case of 13-dytek. MoO<sub>3</sub> used in the reaction probably remained as an amorphous salt dissolved in excess of amine (the sticky fraction surrounding the crystalline carbamates). Preparative studies without the use of MoO<sub>3</sub> and tests using IR spectroscopy indicate the correctness of our hypothesis and confirm the formation of **12-propCO<sub>2</sub>** and **13-dytekCO<sub>2</sub>** monocarbamates in our present studies.

## II. EXPERIMENTAL

All chemicals were purchased from commercial sources and used without further purification. The following amines purchased from Sigma-Aldrich were used for the experiment: 1,2-diaminopropane (12-dap), purity 99%; 1,3-diaminopropane (13-dap), purity 98%; and 1,3-diaminopentane (13-dytek), purity 98%.

## A. Sample preparation

## 1. Synthesis

Five milliliters of diamine 12-dap, 13-dap, and 13-dytek were added to 20 ml of water and the prepared solutions were left to air dry in a beaker covered with filter paper at room

<sup>a)</sup> Author to whom correspondence should be addressed. Electronic mail: lasocha@chemia.uj.edu.pl



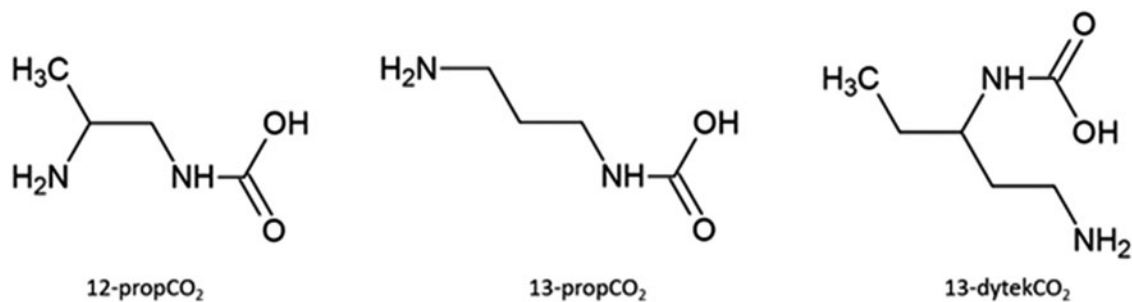


Figure 1. Structural formulas of the obtained compound.

conditions. After 2 months, the obtained white powder products, marked as **12-propCO<sub>2</sub>**, **13-propCO<sub>2</sub>**, and **13-dytekCO<sub>2</sub>**, were thoroughly powdered and investigated by X-ray powder diffraction (XRPD) techniques and elemental analysis.

The elemental analysis showed the following percentage composition for

**12-propCO<sub>2</sub>** C: 40.59%, H: 8.092%, N: 23.57%; calculated values C: 40.67%, H: 8.53%, N: 23.71%;

**13-propCO<sub>2</sub>** C: 39.00%, H: 8.992%, N: 21.69%; calculated values C: 40.67%, H: 8.53%, N: 23.71%;

**13-dytekCO<sub>2</sub>** C: 49.30%, H: 9.627%, N: 18.76%; calculated values C: 49.30%, H: 9.65%, N: 19.16%.

Similar studies were carried out for 1,2-diaminoethane. A mixture of products with known structures deposited in the CSD (Cambridge Structural Database, 2022) were obtained as a result of the reaction, also listed in ICDD PDF-4+ as PDF 00-034-1993 and 00-034-1992.

### B. XRPD measurements and crystallographic studies

**13-propCO<sub>2</sub>** and **13-dytekCO<sub>2</sub>** were thoroughly powdered in a mortar and placed into a flat sample holder. In the

case of **12-propCO<sub>2</sub>**, the obtained product was placed into the thin-walled glass capillary (0.1 mm) filled with the amine 12-dap.

The XRPD measurements were performed at the Faculty of Chemistry, Jagiellonian University, using an X'Pert PRO MPD diffractometer, equipped with a diffracted beam graphite monochromator, a PIXcel 1-D detector and Cu long fine focus ceramic tube (generator setting: 40 kV and 30 mA) at 298 K. The diffraction data were collected over the  $2\theta$  angular range from 3 to 80° (for capillary measurements from 6 to 100°) with a step size of 0.02°. The divergence of the X-ray beam was 0.25°.

### III. RESULTS AND DISCUSSION

The initial analysis of the recorded powder diffraction data was conducted using the PANalytical X'pert HighScore program (Degen et al., 2014) and the PDF-4+ database (Gates-Rector and Blanton, 2019) to verify the purity of the products. For **12-propCO<sub>2</sub>**, no additional phases were found. In the powder data set for **13-propCO<sub>2</sub>** and **13-dytekCO<sub>2</sub>**, some low-intensity reflections were observed. These are most likely related to the formation of

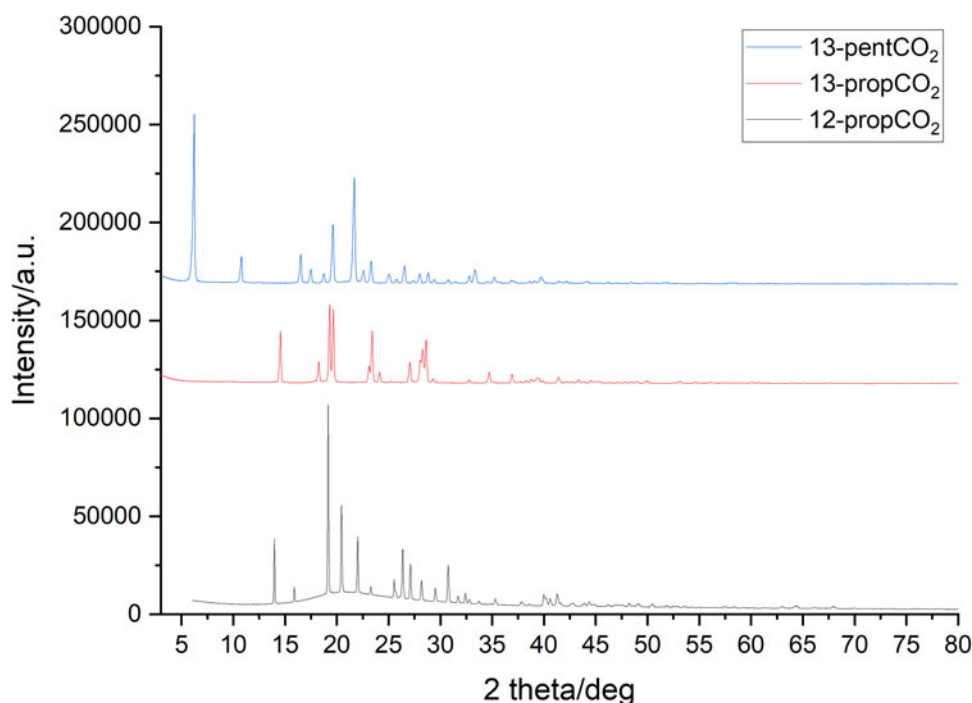


Figure 2. Experimental powder diffraction pattern of the investigated carbamates: **12-propCO<sub>2</sub>** (black line), **13-propCO<sub>2</sub>** (red line), and **13-dytekCO<sub>2</sub>** (blue line).

TABLE I. X-ray powder diffraction data of **12-propCO<sub>2</sub>**.

$2\theta_{\text{obs}}$ (°)	$d_{\text{obs}}$ (Å)	$I_{\text{obs}}$ (%)	$h$	$k$	$l$	$2\theta_{\text{cal}}$ (°)	$d_{\text{cal}}$ (Å)	$\Delta 2\theta$
13.977	6.3310	33.3	−1	1	0	13.987	6.3264	−0.010
15.905	5.5677	7.60	0	1	1	15.913	5.5648	−0.008
19.172	4.6256	100	0	2	0	19.178	4.6243	−0.006
20.457	4.3379	46.1	2	0	0	20.464	4.3364	−0.007
22.022	4.0330	29.3	−2	1	1	22.029	4.0318	−0.007
23.287	3.8167	4.35	−1	2	1	23.293	3.8157	−0.006
25.550	3.4836	8.75	0	0	2	25.550	3.4835	0.000
25.716	3.4615	2.50	−1	1	2	25.713	3.4619	0.003
26.370	3.3771	25.5	−2	0	2	26.369	3.3772	0.001
27.117	3.2857	18.4	1	2	1	27.119	3.2855	−0.002
28.187	3.1634	10.5	−2	2	0	28.189	3.1632	−0.002
29.522	3.0233	7.00	2	1	1	29.525	3.0230	−0.003
30.756	2.9048	19.1	−1	3	0	30.756	2.9048	0.000
31.715	2.8191	3.52	0	3	1	31.714	2.8192	0.001
32.425	2.7589	5.20	−3	1	0	32.421	2.7593	0.004
			1	1	2	32.421	2.7592	0.004
32.819	2.7267	2.10	−2	2	2	32.812	2.7273	0.007
33.737	2.6546	1.58	−3	1	2	33.735	2.6548	0.002
34.839	2.5731	0.50	−3	2	1	34.845	2.5727	−0.005
35.314	2.5396	3.15	−2	3	1	35.313	2.5396	0.001
37.844	2.3754	1.81	−1	3	2	37.815	2.3771	0.029
38.534	2.3345	0.93	2	0	2	38.534	2.3345	0.000
40.006	2.2519	5.22	−4	1	1	39.995	2.2525	0.011
			0	1	3	39.996	2.2524	0.010
40.230	2.2399	3.40	−4	0	2	40.228	2.2400	0.002
40.617	2.2194	3.70	2	3	1	40.611	2.2197	0.006
41.292	2.1847	6.10	−1	2	3	41.294	2.1846	−0.002
41.626	2.1679	1.30	4	0	0	41.620	2.1682	0.006
42.623	2.1195	0.80	3	2	1	42.657	2.1179	−0.034
42.822	2.1101	1.50	−3	3	0	42.850	2.1088	−0.028
			1	3	2	42.850	2.1088	−0.028
43.890	2.0612	1.56	−3	3	2	43.894	2.0610	−0.004
44.372	2.0399	2.32	−2	4	0	44.364	2.0402	0.008
44.729	2.0244	1.02	−3	2	3	44.733	2.0243	−0.004
46.206	1.9631	0.94	4	2	0	46.206	1.9631	0.000
46.818	1.9389	0.73	−4	1	3	46.826	1.9386	−0.008
47.151	1.9260	0.70	0	4	2	47.139	1.9264	0.012
47.349	1.9184	1.70	−2	3	3	47.369	1.9176	−0.020
47.629	1.9077	1.63	−2	4	2	47.626	1.9078	0.003
48.230	1.8854	1.51	1	2	3	48.237	1.8851	−0.007
48.619	1.8712	0.81	3	1	2	48.619	1.8712	0.000
49.109	1.8536	0.67	−4	3	1	49.072	1.8549	0.037
			0	3	3	49.073	1.8549	0.036
			−3	4	1	49.14	1.8526	−0.031
50.466	1.8069	1.51	−5	1	2	50.503	1.8057	−0.037
			−1	1	4	50.504	1.8057	−0.038
51.860	1.7616	0.81	2	1	3	51.852	1.7619	0.008
52.497	1.7417	0.67	0	0	4	52.495	1.7418	0.001
52.823	1.7317	0.64	−2	2	4	52.848	1.7310	−0.025
63.002	1.4742	0.89	5	2	1	62.963	1.4750	0.039
			3	2	3	62.964	1.4750	0.038
			−6	1	3	63.002	1.4742	0.000
64.348	1.4466	1.34	−4	5	1	64.334	1.4469	0.014
			0	5	3	64.335	1.4469	0.013
67.926	1.3788	1.14	2	2	4	67.882	1.3796	0.044

competing phases that differ in hydration or the progressing carbonization process. In order to obtain the unit cell parameters and the space group, the XRPD patterns of **12-propCO<sub>2</sub>**, **13-propCO<sub>2</sub>**, and **13-dytekCO<sub>2</sub>** were indexed using the N-TREOR program built in the program EXPO2014 (Altomare et al., 2013). For each compound, the space group was determined by the reflection conditions derived from the indexed reflections. The observed integrated intensities were extracted by the Le Bail method using the program

EXPO2004. The monoclinic cell  $a = 9.3005$ ,  $b = 9.2471$ ,  $c = 7.4743$  Å, and  $\beta = 111.1854^\circ$ ,  $V = 599.53$  Å<sup>3</sup>, with figures of merit  $M_{20} = 44$  and  $F_{20} = 56$  (0.005329, 68) (De Wolf, 1968; Smith and Snyder, 1979) were found for **12-propCO<sub>2</sub>**. All lines were indexed. The analysis indicated the most probable space group as *Ia* (FoM = 0.536). For **13-propCO<sub>2</sub>**, the solution with highest figures of merit  $M_{20} = 64$  and  $F_{20} = 98$  (0.007339, 28) indexed all reflections in the monoclinic system with unit cell parameters  $a = 5.0003$ ,  $b = 12.1937$ ,

TABLE II. X-ray powder diffraction data of **13-propCO<sub>2</sub>**.

$2\theta_{\text{obs}}$ (°)	$d_{\text{obs}}$ (Å)	$I_{\text{obs}}$ (%)	$h$	$k$	$l$	$2\theta_{\text{cal}}$ (°)	$d_{\text{cal}}$ (Å)	$\Delta 2\theta$
14.520	6.1005	63	0	2	0	14.510	6.1047	0.010
17.839	4.9721	0.85	1	0	0	17.830	4.9748	0.009
18.220	4.8692	27.5	0	0	1	18.219	4.8694	0.001
19.279	4.6039	96	1	1	0	19.266	4.6071	0.013
19.638	4.5206	100	0	1	1	19.628	4.5229	0.011
23.079	3.8538	19.0	1	2	0	23.063	3.8565	0.016
23.378	3.8052	72	0	2	1	23.368	3.8068	0.010
24.099	3.6929	15.0	-1	0	1	24.094	3.6937	0.005
25.199	3.5342	2.81	-1	1	1	25.190	3.5355	0.009
27.020	3.300	31.3	1	0	1	27.026	3.2993	-0.005
28.040	3.1822	31.9	1	1	1	28.015	3.1850	0.025
28.240	3.1601	51.2	-1	2	1	28.239	3.1603	0.001
28.600	3.1213	59.7	0	3	1	28.586	3.1227	0.014
29.261	3.0522	3.70	0	4	0	29.260	3.0523	0.001
32.758	2.7339	5.70	-1	3	1	32.743	2.7351	0.015
34.700	2.5852	18.4	0	4	1	34.686	2.5862	0.014
34.960	2.5666	1.84	1	3	1	35.012	2.5629	-0.052
36.120	2.4868	0.95	2	0	0	36.111	2.4874	0.009
36.899	2.4361	13.5	2	1	0	36.879	2.4373	0.020
			0	0	2	36.920	2.4347	-0.021
37.698	2.3863	1.52	0	1	2	37.674	2.3877	0.024
38.260	2.3525	3.51	-1	4	1	38.253	2.3529	0.008
38.761	2.3232	5.74	-2	0	1	38.756	2.3235	0.005
39.380	2.2881	17.4	-1	0	2	39.329	2.2910	0.051
39.841	2.2627	4.24	0	2	2	39.863	2.2615	-0.022
41.379	2.1821	11.0	0	5	1	41.365	2.1828	0.014
42.120	2.1454	2.17	-1	2	2	42.130	2.1449	-0.010
			2	3	0	42.599	2.1224	0.020
42.619	2.1214	1.76	2	0	1	42.636	2.1206	-0.017
43.299	2.0896	6.44	0	3	2	43.305	2.0894	-0.006
			2	1	1	43.306	2.0893	-0.016
43.825	2.0658	2.11	1	1	2	43.830	2.0656	-0.005
44.500	2.0360	4.76	-1	5	1	44.478	2.0370	0.022
			0	6	0	44.526	2.0349	-0.026
44.921	2.0179	2.19	-2	3	1	44.923	2.0178	-0.003
45.281	2.0027	4.72	2	2	1	45.269	2.0032	0.012
46.279	1.9618	1.29	1	5	1	46.255	1.9628	0.024
47.100	1.9295	1.85	2	4	0	47.134	1.9282	-0.034
47.780	1.9036	2.53	0	4	2	47.787	1.9034	-0.007
48.400	1.8807	2.54	2	3	1	48.402	1.8806	-0.002
48.900	1.8626	3.50	1	3	2	48.882	1.8633	0.018
49.898	1.8277	6.34	-2	1	2	49.945	1.8261	-0.047
			2	5	0	52.517	1.7425	0.043
52.560	1.7412	2.93	2	4	1	52.549	1.7415	0.011
53.080	1.7254	4.38	0	5	2	53.121	1.7241	-0.041
			-2	5	1	54.517	1.6832	0.042
54.559	1.6820	3.67	-2	3	2	54.568	1.6818	-0.009
55.961	1.6432	3.45	3	1	0	55.962	1.6432	-0.001
			0	7	1	56.004	1.6420	-0.043
57.138	1.6121	2.59	-3	1	1	57.131	1.6123	0.007
57.978	1.5907	3.81	1	5	2	57.993	1.5904	-0.015
59.981	1.5423	2.94	1	7	1	59.995	1.5420	-0.014
60.697	1.5258	1.67	0	8	0	60.682	1.5262	0.015
61.483	1.5082	1.41	0	3	3	61.507	1.5077	-0.023
63.901	1.4568	2.06	3	4	0	63.888	1.4571	0.014
			0	8	1	63.926	1.4563	-0.026
65.001	1.4348	1.35	-2	0	3	64.981	1.4352	0.020

$c = 4.8968 \text{ \AA}$ ,  $\beta = 96.47^\circ$ , and  $V = 296.67 \text{ \AA}^3$ .  $P2_1$  was indicated as the most probable space group (FoM = 0.889). For **13-dytekCO<sub>2</sub>**, the hexagonal unit cell with the highest figures of merit  $M_{20} = 53$  and  $F_{20} = 90$  (0.008288, 27), unit cell parameters  $a = 28.3506$ ,  $b = 28.3506$ ,  $c = 5.1702 \text{ \AA}$ , and  $V = 3598.87 \text{ \AA}^3$  were obtained. The analysis indicated the

most probable space group as  $R\bar{3}$  (FoM = 0.282). The structural formulas of the investigated compounds are illustrated in [Figure 1](#).

Experimental powder diffraction patterns are presented in [Figure 2](#). XRPD data of the studied compounds are shown in [Tables I–III](#). The structural data for all three compounds are

TABLE III. X-ray powder diffraction data of 13-dytekCO<sub>2</sub>.

$2\theta_{\text{obs}}$ (°)	$d_{\text{obs}}$ (Å)	$I_{\text{obs}}$ (%)	$h$	$k$	$l$	$2\theta_{\text{cal}}$ (°)	$d_{\text{cal}}$ (Å)	$\Delta 2\theta$
6.238	14.1684	100	1	1	0	6.230	14.1872	0.008
10.799	8.1931	16.0	3	0	0	10.801	8.1912	-0.003
12.479	7.0932	0.79	2	2	0	12.479	7.0933	0.000
16.538	5.3605	17.0	4	1	0	16.532	5.3623	0.005
17.499	5.0680	8.59	1	0	1	17.522	5.0615	-0.022
18.759	4.7305	5.46	3	3	0	18.765	4.7289	-0.006
19.638	4.5207	37.7	2	1	1	19.645	4.5191	-0.008
21.698	4.0960	74	6	0	0	21.700	4.0955	-0.003
22.580	3.9379	7.82	5	2	0	22.598	3.9348	-0.018
23.322	3.8143	14.7	3	2	1	23.340	3.8113	-0.018
25.061	3.5533	7.63	4	4	0	25.109	3.5467	-0.047
25.778	3.4561	2.54	2	4	1	25.783	3.4555	-0.004
26.539	3.3587	11.8	5	1	1	26.550	3.3574	-0.011
27.399	3.2552	1.52	7	1	0	27.403	3.2548	-0.004
28.019	3.1846	6.38	4	3	1	28.026	3.1838	-0.007
28.839	3.0959	6.83	6	3	0	28.839	3.0959	0.000
29.421	3.0359	2.68	1	6	1	29.434	3.0346	-0.013
30.779	2.9051	2.77	7	0	1	30.783	2.9047	-0.004
31.442	2.8452	1.49	6	2	1	31.438	2.8456	0.004
32.799	2.7306	5.32	9	0	0	32.803	2.7303	-0.004
33.342	2.6873	11.3	5	4	1	33.333	2.6881	0.009
34.540	2.5968	1.06	2	7	1	34.546	2.5964	-0.006
35.240	2.5468	5.00	7	4	0	35.223	2.5480	0.017
35.720	2.5137	1.15	8	1	1	35.722	2.5136	-0.002
36.860	2.4385	2.80	7	3	1	36.865	2.4382	-0.005
37.179	2.4184	1.03	3	1	2	37.184	2.4180	-0.005
38.620	2.3314	1.15	10	1	0	38.604	2.3323	0.016
39.060	2.3061	1.66	1	9	1	39.065	2.3058	-0.005
39.680	2.2715	8.13	9	3	0	39.676	2.2717	0.004
41.460	2.1780	2.41	3	4	2	41.457	2.1782	0.003
42.180	2.1425	2.10	5	7	1	42.184	2.1423	-0.004
42.679	2.1186	0.52	8	4	1	42.686	2.1182	-0.007
43.460	2.0823	0.31	0	7	2	43.462	2.0822	-0.002
44.000	2.0580	1.45	2	6	2	43.952	2.0601	0.048
				11	1	44.162	2.0508	0.038
44.200	2.0491	1.85	12	0	0	44.232	2.0477	-0.032
44.701	2.0273	0.66	11	2	0	44.715	2.0267	-0.014
46.158	1.9667	1.32	10	4	0	46.140	1.9674	0.017
47.041	1.9318	0.94	10	3	1	47.005	1.9332	0.036
			6	8	1	48.378	1.8815	0.042
48.420	1.8800	1.18	9	6	0	48.443	1.8791	-0.023
48.822	1.8654	0.45	9	5	1	48.829	1.8652	-0.007
51.493	1.7748	2.72	13	0	1	51.474	1.7754	0.019
			8	8	0	51.536	1.7734	-0.043
51.858	1.7631	1.90	12	2	1	51.905	1.7616	-0.047
54.853	1.6737	1.19	7	9	1	54.856	1.6736	-0.004
58.058	1.5887	1.41	6	0	3	58.043	1.5891	0.015
			13	3	1	58.104	1.5876	-0.046
58.563	1.5762	1.45	9	9	0	58.559	1.5763	0.004
60.797	1.5236	0.49	7	1	3	60.795	1.5236	0.002

TABLE IV. Crystal data for the investigated compounds.

	12-propCO <sub>2</sub>	13-propCO <sub>2</sub>	13-dytekCO <sub>2</sub>
Lattice parameters			
$a$ (Å)	9.3033 (7)	5.0065 (10)	28.374 (3)
$b$ (Å)	9.2485 (7)	12.2093 (23)	—
$c$ (Å)	7.4735 (7)	4.9006 (10)	5.1726 (9)
$\beta$ (°)	111.214 (7)	96.457 (18)	—
Volume (Å <sup>3</sup> )	599.46	297.65	3606.53
$Z$	4	2	18
Space group	$Ia$	$P2_1$	$R-3$
Crystal system	Monoclinic	Monoclinic	Trigonal
$M_{20}$	44	64	53
$F_{20}$	56 (0.005329, 68)	98 (0.007339, 28)	90 (0.008288, 27)

given in Table IV. The diffraction patterns for all compounds are available in SI in cif and xy format.

#### IV. CONCLUSION

On the basis of the conducted research, it was established that all the obtained compounds: **12-propCO<sub>2</sub>**, **13-propCO<sub>2</sub>**, and **13-dytekCO<sub>2</sub>** are monocarbamates. Unit cell parameters and probable space groups were determined. Structural studies are the subject of ongoing research.

Monocarbamates can be formed as unexpected, unwanted products of synthetic reactions carried out in the presence of moisture and air, in which amines are used as substrates.

#### V. DEPOSITED DATA

CIF and xy data files for the three reported compounds were deposited with ICDD. You can request these data by contacting ICDD at [pdj@icdd.com](mailto:pdj@icdd.com).

#### Supplementary material

The supplementary material for this article can be found at <https://doi.org/10.1017/S0885715623000404>.

#### REFERENES

Altomare, A., C. Cuocci, C. Giacobozzo, A. Moliterni, R. Rizzi, N. Corriero, and A. Falcicchio. 2013. "EXPO2013: A Kit of Tools for Phasing Crystal

Structures from Powder Data." *Journal of Applied Crystallography* 46 (4): 1231–35. doi:10.1107/S0021889813013113.

Degen, T., M. Sadki, E. Bron, U. König, and G. Nénert. 2014. "The HighScore Suite." *Powder Diffraction* 29 (S2): S13–18. doi:10.1017/S0885715614000840.

De Wolf, P. M. 1968. "A Simplified Criterion for the Reliability of a Powder Pattern Indexing." *Journal of Applied Crystallography* 1 (2): 108–13. doi:10.1107/S002188986800508X.

Gates-Rector, S., and T. Blanton. 2019. "The Powder Diffraction File: A Quality Materials Characterization Database." *Powder Diffraction* 34 (4): 352–60. doi:10.1017/S0885715619000812.

Gonzalez, G. A., M. Oszejca, K. Luberd-Durnaś, and W. Lasocha. 2019. "Synthesis, Crystal Structure Solution and Characterization of Two Organic–Inorganic Hybrid Layered Materials Based on Metal Sulfates and 1,4-Phenylenediamine." *Acta Crystallographica Section C Structural Chemistry* 75 (11): 1502–8. doi:10.1107/S205322961901180X.

Leszczyński, M. K., D. Kornacki, M. Terlecki, I. Justyniak, G. I. Miletic, I. Halasz, P. Bernatowicz, V. Szejko, and J. Lewiński. 2022. "Mechanochemical Vs Wet Approach for Directing CO<sub>2</sub> Capture Toward Various Carbonate and Bicarbonate Networks." *ACS Sustainable Chemistry Engineering* 10 (14): 4373–80. doi:10.1021/acsschemeng.1c08402.

Luberd-Durnaś, K., B. Gawel, M. Łoś, and W. Łasocha. 2011. "Synthesis and Characterization of Inorganic–Organic ZnS(Aminopropane)<sub>n</sub> Composite Materials." *Crystal Research and Technology* 46 (12): 1283–90. doi:10.1002/crat.201100380.

Smith, G. S., and R. L. Snyder. 1979. "F<sub>N</sub>: A Criteria for Rating Powder Diffraction Patterns and Evaluating the Reliability of Powder-Pattern Indexing." *Journal of Applied Crystallography* 12 (1): 60–65. doi:10.1107/S002188987901178X

Zhou, H., F. Shi, X. Tian, Q. Zhang, and Y. Deng. 2007. "Synthesis of Carbamates from Aliphatic Amines and Dimethyl Carbonate Catalyzed by Acid Functional Ionic Liquids." *Journal of Molecular Catalysis A: Chemical* 271 (1–2): 89–92. doi:10.1016/j.molcata.2007.02.017.