X-ray powder diffraction data for 3-amino-5-(aminocarbonyl)-2,4,6-triiodobenzoic acid methanol solvate

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X-ray powder diffraction data, unit-cell parameters, and space group for a $C_9H_9I_3N_2O_4$ are presented [$a = 17.000$ (1) Å, $b = 13.896$ (1) Å, $c = 12.597$ (1) Å, unit-cell volume $V = 2975.9$ Å^3, MW = 589.89, T_m (melting point) = 300(1) °C, $\rho_c = 2.641$ g cm^{-3}, $\rho_m = 2.66(3)$ g cm^{-3}, $Z = 8$, space group *Pbca*]. All measured lines were indexed and are consistent with the *Pbca* space group. No detectable impurities were observed. © *2017 International Centre for Diffraction Data.*
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Key words: X-ray powder diffraction, radiocontrast agents

I. INTRODUCTION

3-Amino-5-(aminocarbonyl)-2,4,6-triiodobenzoic acid is currently used as a precursor of iosimenol, a non-ionic radiocontrast agent with both low osmolality and viscosity and good water solubility (Sovak *et al.*, 2004; Bailey *et al.*, 2013). In order to produce iosimenol in commercial quantities, its manufacture process must be economic. Such process requires efficient purification techniques, for assuring high yields and high purities in each synthetic step. However, the use of such techniques needs to be cost-effective, and crystallization seems to be the best method. In this manner, the structure of single iosimenol precursors and accompanying impurities is seeked in order to design superior purification techniques. Thus, we present data for the title compound crystallizing as a methanol solvate, as shown in Figure 1.

We have inspected the CSD database (Allen, 2002) and the PDF4+ database (ICDD, 2015) and have not found any entry for this organic substance $C_9H_9I_3N_2O_4$ in the mentioned databases. Therefore, we have decided to characterize this compound by X-ray powder diffraction (XRD) technique.

II. EXPERIMENTAL

A. Synthesis

The title compound was prepared by reacting 3-amino-5-(aminocarbonyl) benzoic acid and sodium iodine dichloride or iodine monochloride in an aqueous solvent, under acidic conditions, at a temperature of 65–85 °C. The crude product was then recrystallized twice from a refluxing mixture of methanol/water.

B. Density measurement

For the title compound, experimental density was evaluated as an average of 10 measurements in 50 ml pycnometer, using *n*-heptane as a liquid, resulting in a value of $\rho_m = 2.66$ (3) g cm^{-3}.

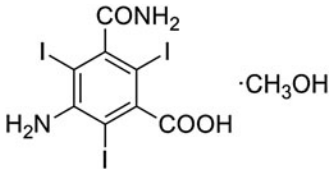

Figure 1. Structural formula of 3-amino-5-(aminocarbonyl)-2,4,6-triiodobenzoic acid methanol solvate.

C. Specimen preparation

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

D. 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 80°2θ with a step size of 0.013°2θ and a counting time of 118.32 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 I). The *d*-values were calculated using Cu$K\alpha_1$ radiation ($\lambda = 1.5406$ Å).

III. RESULTS AND DISCUSSION

The experimental powder diffraction pattern is depicted in Figure 2. Automatic indexing of results obtained using

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TABLE I. Indexed X-ray powder diffraction data for $C_9H_9I_3N_2O_4$. Only the peaks with I_{rel} of 1 or greater are presented [$a = 17.000$ (1) Å, $b = 13.896$ (1) Å, $c = 12.597$ (1) Å, unit-cell volume $V = 2975.9$ Å^3, $Z = 8$, space group *Pbca*]. All lines were indexed and are consistent with the *Pbca* 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_{cal} (Å)	$\Delta 2\theta$
10.438	8.4683	19	2	0	0	10.399	8.5002	−0.039
12.228	7.2323	45	2	1	0	12.196	7.2512	−0.032
12.769	6.9269	46	0	2	0	12.730	6.9481	−0.039
14.126	6.2645	3	2	1	1	14.081	6.2843	−0.045
14.582	6.0699	15	0	2	1	14.548	6.0840	−0.034
15.020	5.8936	34	1	0	2	14.989	5.9060	−0.032
16.324	5.4256	5	1	1	2	16.295	5.4355	−0.030
16.500	5.3683	3	2	2	0	16.465	5.3796	−0.035
17.947	4.9385	18	2	2	1	17.915	4.9473	−0.032
18.673	4.7482	18	2	1	2	18.646	4.7550	−0.027
19.035	4.6586	13	0	2	2	19.003	4.6664	−0.032
19.742	4.4934	4	1	2	2	19.713	4.5000	−0.029
20.915	4.2440	15	4	0	0	20.884	4.2501	−0.030
21.097	4.2077	100	1	3	1	21.076	4.2119	−0.021
21.438	4.1417	50	3	2	1	21.411	4.1467	−0.026
21.867	4.0612	24	4	1	0	21.851	4.0643	−0.017
22.051	4.0278	12	3	1	2	22.031	4.0315	−0.020
22.739	3.9074	76	1	1	3	22.715	3.9115	−0.024
22.991	3.8652	12	4	1	1	22.975	3.8679	−0.017
24.551	3.6230	32	4	2	0	24.533	3.6256	−0.018
24.716	3.5992	22	3	2	2	24.695	3.6023	−0.022
25.285	3.5195	83	4	0	2	25.259	3.5230	−0.026
25.578	3.4799	10	4	2	1	25.546	3.4841	−0.032
25.637	3.4719	9	0	4	0	25.621	3.4741	−0.016
26.083	3.4135	26	4	1	2	26.072	3.4150	−0.011
26.628	3.3450	27	0	4	1	26.595	3.3490	−0.033
27.194	3.2766	31	3	1	3	27.178	3.2784	−0.016
27.752	3.2120	7	2	4	0	27.718	3.2158	−0.034
27.922	3.1928	16	5	1	1	27.905	3.1947	−0.016
28.402	3.1399	27	4	2	2	28.381	3.1422	−0.021
28.651	3.1132	19	2	4	1	28.626	3.1159	−0.026
28.826	3.0947	15	1	0	4	28.809	3.0965	−0.017
29.183	3.0577	8	1	3	3	29.159	3.0601	−0.024
29.366	3.0391	29	4	3	1	29.365	3.0391	−0.001
29.847	2.9911	1	5	0	2	29.839	2.9919	−0.008
30.080	2.9684	1	5	2	1	30.085	2.9680	0.004
30.251	2.9521	8	2	0	4	30.241	2.9530	−0.010
30.594	2.9198	5	4	1	3	30.588	2.9203	−0.006
31.016	2.8810	30	3	4	1	30.992	2.8832	−0.024
31.224	2.8623	27	2	4	2	31.204	2.8641	−0.020
31.616	2.8277	8	1	2	4	31.609	2.8283	−0.007
31.903	2.8029	12	4	3	2	31.889	2.8041	−0.015
32.600	2.7445	2	4	2	3	32.605	2.7442	0.004
32.832	2.7257	6	3	3	3	32.815	2.7270	−0.017
32.926	2.7181	5	2	2	4	32.931	2.7177	0.005
33.012	2.7112	5	6	1	1	33.012	2.7112	0.000
33.313	2.6874	13	4	4	0	33.283	2.6898	−0.030
33.425	2.6786	22	5	3	1	33.430	2.6783	0.005
33.895	2.6426	23	2	5	0	33.908	2.6416	0.013
34.073	2.6292	10	4	4	1	34.056	2.6305	−0.018
34.696	2.5834	10	6	0	2	34.688	2.5840	−0.008
34.816	2.5748	8	1	3	4	34.823	2.5743	0.007
34.913	2.5679	8	6	2	1	34.903	2.5685	−0.009
35.039	2.5589	12	3	2	4	35.035	2.5591	−0.004
35.454	2.5299	3	4	0	4	35.448	2.5303	−0.006
35.731	2.5109	3	4	3	3	35.739	2.5103	0.008
36.054	2.4891	3	4	1	4	36.051	2.4893	−0.003
36.349	2.4696	13	5	2	3	36.346	2.4698	−0.003
36.637	2.4508	3	1	1	5	36.604	2.4530	−0.033
36.686	2.4477	3	3	5	1	36.685	2.4477	−0.001
37.126	2.4197	2	3	4	3	37.117	2.4203	−0.009
37.704	2.3839	1	5	4	1	37.670	2.3860	−0.034
37.803	2.3779	3	4	2	4	37.809	2.3775	0.007
37.862	2.3743	3	6	3	1	37.871	2.3738	0.009
37.959	2.3685	18	0	2	5	37.960	2.3684	0.001
38.270	2.3499	6	7	1	1	38.263	2.3503	−0.007
38.364	2.3444	3	1	2	5	38.341	2.3458	−0.024
38.862	2.3155	2	6	1	3	38.856	2.3158	−0.006
38.954	2.3102	3	5	0	4	38.951	2.3104	−0.003
39.232	2.2945	7	5	3	3	39.220	2.2952	−0.013
39.341	2.2884	5	4	5	1	39.359	2.2874	0.018
39.473	2.2811	5	2	2	5	39.465	2.2815	−0.008
39.652	2.2712	4	3	1	5	39.653	2.2711	0.001
39.743	2.2662	4	7	0	2	39.746	2.2660	0.004
39.907	2.2572	2	1	6	1	39.899	2.2577	−0.009
40.333	2.2344	3	2	6	0	40.329	2.2346	−0.004
41.096	2.1946	16	1	3	5	41.096	2.1946	0.000
41.289	2.1848	9	3	2	5	41.281	2.1852	−0.009
41.521	2.1731	5	0	6	2	41.509	2.1737	−0.012
41.758	2.1614	4	6	4	1	41.723	2.1631	−0.035
41.877	2.1555	11	1	6	2	41.863	2.1562	−0.014
42.099	2.1446	6	3	5	3	42.090	2.1451	−0.010
42.582	2.1214	2	5	5	1	42.589	2.1211	0.007
42.947	2.1042	14	5	4	3	42.970	2.1032	0.023
43.046	2.0996	10	0	0	6	43.050	2.0994	0.004
43.638	2.0725	6	8	1	1	43.649	2.0720	0.011
43.732	2.0683	7	1	5	4	43.731	2.0683	−0.001
43.880	2.0616	4	3	3	5	43.883	2.0615	0.003
44.544	2.0324	5	8	2	0	44.551	2.0321	0.007
44.642	2.0282	4	0	6	3	44.647	2.0280	0.005
45.005	2.0127	10	7	2	3	45.016	2.0122	0.012
45.081	2.0095	11	0	2	6	45.076	2.0097	−0.005
45.223	2.0035	10	5	1	5	45.233	2.0031	0.010
45.715	1.9831	4	2	4	5	45.711	1.9832	−0.004
46.067	1.9687	3	3	0	6	46.068	1.9687	0.002
46.322	1.9585	2	6	5	1	46.286	1.9599	−0.036
46.982	1.9325	5	2	7	0	46.965	1.9332	−0.018
47.217	1.9234	10	7	0	4	47.224	1.9232	0.007
47.350	1.9183	9	3	4	5	47.333	1.9190	−0.017
47.450	1.9145	10	7	3	3	47.454	1.9144	0.004
47.587	1.9093	12	3	6	3	47.585	1.9094	−0.002
47.996	1.8940	4	3	2	6	47.993	1.8941	−0.003
48.307	1.8825	3	4	0	6	48.313	1.8823	0.006
49.084	1.8545	15	1	6	4	49.081	1.8546	−0.003
49.532	1.8388	3	4	4	5	49.534	1.8387	0.001
49.761	1.8309	2	4	6	3	49.778	1.8303	0.016
50.156	1.8174	4	6	2	5	50.162	1.8172	0.006
50.303	1.8124	4	8	4	0	50.292	1.8128	−0.011
50.544	1.8044	4	9	2	1	50.555	1.8040	0.011
50.705	1.7990	6	4	7	0	50.715	1.7986	0.010
50.872	1.7935	9	6	6	0	50.880	1.7932	0.008
51.421	1.7756	4	6	6	1	51.430	1.7753	0.009
51.506	1.7729	5	3	5	5	51.506	1.7729	0.000
52.478	1.7423	3	8	4	2	52.485	1.7421	0.008
52.703	1.7354	3	7	1	5	52.722	1.7348	0.019
53.533	1.7104	3	3	7	3	53.515	1.7109	−0.017
53.843	1.7013	7	2	8	0	53.824	1.7019	−0.019
54.703	1.6766	3	7	5	3	54.700	1.6767	−0.003
54.814	1.6734	3	8	5	1	54.824	1.6732	0.010
55.068	1.6663	4	1	8	2	55.063	1.6665	−0.004
55.812	1.6459	4	2	3	7	55.820	1.6456	0.008
55.939	1.6424	4	2	8	2	55.920	1.6429	−0.019
56.091	1.6383	3	6	2	6	56.058	1.6392	−0.033
56.186	1.6358	4	7	3	5	56.185	1.6358	−0.001
57.356	1.6052	2	0	8	3	57.359	1.6051	0.002
57.657	1.5975	1	10	2	2	57.664	1.5973	0.007
58.246	1.5827	2	4	6	5	58.258	1.5824	0.012
58.919	1.5663	1	8	5	3	58.918	1.5663	−0.001

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TABLE I. Continued

$2\theta_{obs}$ (°)	d_{obs} (Å)	I_{obs}	h	k	l	$2\theta_{cal}$ (°)	d_{cal} (Å)	$\Delta 2\theta$
59.103	1.5618	2	7	4	5	59.103	1.5618	0.000
59.268	1.5579	2	4	8	2	59.264	1.5580	−0.004
59.463	1.5532	2	1	7	5	59.483	1.5527	0.020
59.648	1.5489	2	1	6	6	59.641	1.5490	−0.006
59.846	1.5442	2	3	8	3	59.839	1.5443	−0.007
60.214	1.5356	2	0	2	8	60.214	1.5356	0.000
60.432	1.5306	2	2	6	6	60.456	1.5301	0.024
60.667	1.5253	1	7	7	1	60.647	1.5257	−0.020
61.023	1.5172	2	3	0	8	61.028	1.5171	0.005
61.127	1.5149	2	1	8	4	61.124	1.5149	−0.003
61.700	1.5022	4	5	8	2	61.698	1.5022	−0.003
62.526	1.4843	3	10	4	2	62.539	1.4840	0.013
62.693	1.4807	2	6	8	0	62.686	1.4809	−0.007
62.885	1.4767	2	9	2	5	62.881	1.4768	−0.005
63.296	1.4681	2	9	4	4	63.294	1.4681	−0.002
63.653	1.4607	2	4	6	6	63.652	1.4607	−0.001
64.100	1.4516	1	5	8	3	64.105	1.4515	0.004
64.480	1.4440	5	1	9	3	64.482	1.4439	0.002
64.625	1.4411	5	8	7	1	64.621	1.4411	−0.003
65.252	1.4287	1	5	0	8	65.248	1.4288	−0.004
65.404	1.4258	2	9	6	2	65.402	1.4258	−0.002
65.517	1.4236	2	10	3	4	65.518	1.4236	0.001
65.604	1.4219	2	2	7	6	65.594	1.4221	−0.011
65.717	1.4197	2	11	2	3	65.716	1.4198	−0.002
65.883	1.4166	2	12	0	0	65.876	1.4167	−0.008
66.006	1.4142	2	4	9	2	66.008	1.4142	0.002
66.571	1.4036	2	11	4	1	66.587	1.4033	0.017
66.672	1.4017	2	2	6	7	66.680	1.4016	0.007
66.944	1.3967	2	6	8	3	66.948	1.3966	0.004
67.200	1.3920	2	6	4	7	67.208	1.3918	0.008

Continued

TABLE I. Continued

$2\theta_{obs}$ (°)	d_{obs} (Å)	I_{obs}	h	k	l	$2\theta_{cal}$ (°)	d_{cal} (Å)	$\Delta 2\theta$
67.400	1.3883	3	5	8	4	67.395	1.3884	−0.005
67.730	1.3823	2	9	6	3	67.737	1.3822	0.007
68.353	1.3713	3	8	7	3	68.360	1.3711	0.007
68.569	1.3675	3	1	2	9	68.561	1.3676	−0.008
68.797	1.3635	3	6	6	6	68.795	1.3635	−0.002
68.945	1.3609	2	11	2	4	68.967	1.3606	0.021
69.698	1.3481	1	6	9	1	69.701	1.3480	0.004
70.227	1.3392	1	10	6	2	70.231	1.3391	0.004
71.184	1.3235	1	2	3	9	71.188	1.3235	0.004
71.261	1.3223	2	2	8	6	71.275	1.3220	0.014
71.914	1.3119	1	12	4	0	71.917	1.3118	0.003
72.296	1.3059	1	10	4	5	72.297	1.3059	0.001
72.529	1.3023	1	0	6	8	72.536	1.3021	0.007
73.530	1.2870	1	2	6	8	73.520	1.2871	−0.011
73.718	1.2842	1	12	4	2	73.711	1.2843	−0.006
74.028	1.2796	1	6	4	8	74.026	1.2796	−0.001
74.691	1.2698	1	12	2	4	74.664	1.2702	−0.027
74.966	1.2658	1	9	6	5	74.978	1.2657	0.012
75.346	1.2604	1	5	10	2	75.352	1.2603	0.006
75.595	1.2569	1	10	5	5	75.595	1.2569	0.000
75.846	1.2533	1	9	5	6	75.846	1.2533	0.001
76.427	1.2452	1	6	9	4	76.424	1.2453	−0.003
76.756	1.2407	1	1	9	6	76.770	1.2405	0.014
77.474	1.2310	1	10	1	7	77.481	1.2309	0.007
77.746	1.2274	1	3	11	1	77.762	1.2272	0.017
78.008	1.2239	2	6	10	2	78.011	1.2239	0.003
79.068	1.2101	1	6	8	6	79.069	1.2101	0.001
79.336	1.2067	1	1	11	3	79.340	1.2067	0.004
79.760	1.2014	1	13	4	2	79.757	1.2014	−0.003

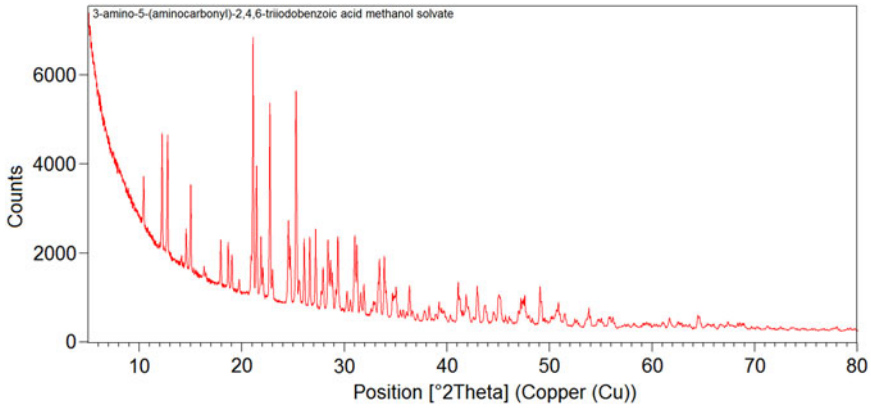

Figure 2. (Color online) X-ray powder diffraction pattern of $C_9H_9I_3N_2O_4$ using Cu$K\alpha$ radiation (λ = 1.5418 Å).

DICVOL04 (Boultif and Louër, 2004) show that title compound $C_9H_9I_3N_2O_4$ is orthorombic with space group *Pbca* and unit-cell parameters: a = 17.000 (1) Å, b = 13.896 (1) Å, c = 12.597 (1) Å, unit-cell volume V = 2975.9 Å^3, and Z = 8. The figures of merits are F_{20} = 70.5(0.0053, 53) (Smith and Snyder, 1979), and M_{20} = 28.7 (de Wolff, 1968). All measured lines were indexed and are consistent with the *Pbca* space group.

SUPPLEMENTARY MATERIAL

The supplementary material for this article can be found at https://doi.org/10.1017/S0885715617000598.

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