X-ray powder diffraction data of the antifungal agents, clotrimazole and fluconazole monohydrate

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Clotrimazole ($C_{22}H_{17}ClN_2$, 1-[(2-chlorophenyl)(diphenyl)methyl]-1*H*-imidazole) and fluconazole ($C_{13}H_{12}F_2N_6$, 2-(2,4-difluorophenyl)-1,3-*bis*(1*H*-1,2,4-triazole-1-yl)propan-2-ol) are two active pharmaceutical ingredients commonly used in the treatment and prevention of superficial and systemic fungal infections. The X-ray powder diffraction data and the unit cell parameters of anhydrous clotrimazole [Triclinic, $P\bar{1}$, $a = 8.776(1)$ Å, $b = 10.571(2)$ Å, $c = 10.622(3)$ Å, $\alpha = 114.08(2)°$, $\beta = 96.87(2)°$, $\gamma = 97.61(2)°$, $V = 875.2(2)$ Å^3, $Z = 2$] and monohydrated fluconazole [Triclinic, $P\bar{1}$, $a = 5.6353(4)$ Å, $b = 11.753(1)$ Å, $c = 12.326(1)$ Å, $\alpha = 71.220(8)°$, $\beta = 79.896(9)°$, $\gamma = 84.35(1)°$, $V = 760.13(9)$ Å^3, $Z = 2$] are reported. © *2013 International Centre for Diffraction Data*.
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Key words: clotrimazole, fluconazole, X-ray diffraction powder data

I. INTRODUCTION

Clotrimazole (an imidazole) and fluconazole (a triazole) are common antifungal agents which belong to the azole family of pharmaceutical compounds (see Figure 1). Both active pharmaceutical ingredients (APIs) can be administered systemically and topically. The azoles exhibit the same wide antifungal spectrum and mechanism of action. They stop fungi from producing ergosterol, an essential component of fungal cell membranes. If ergosterol synthesis is completely or partially inhibited, the fungal cell is unable to construct an intact cell membrane, and dies. Most of the azole formulations are available as over-the-counter medications in various forms: tablets, lozenges, creams, ear drops, etc. Fungal infections are one of the most common causes of skin disease. They are usually treated with topical and oral imidazoles and triazoles among other types of antifungal agents. In particular, *tinea pedis* (athlete's foot) and *tinea cruris* (jock itch) are commonly treated topically with azoles. The latest developments in systemic antifungals are based on triazoles (Bennet, 2006).

A search of the PDF-4/Organics 2013 database lead to three reports related to clotrimazole: PDF 00-035-1915, 00-051-2002, and 02-071-0743 (ICDD, 2013). The first two contain low-quality experimental patterns (Hoogerheide and Wyka, 1982 and Mayer, 1999, respectively). The third (Song and Shin, 1998) corresponds to a calculated pattern using the crystallographic information contained in the Cambridge Structural Database (CSD, V 5.33) (Allen, 2002). This is the only report included in the CSD for this important pharmaceutical compound (Refcode PUVRIH).

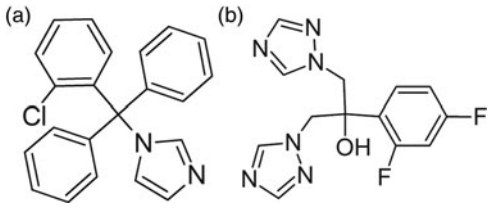

Figure 1. Chemical structure of (a) clotrimazole and (b) fluconazole.

For fluconazole, there are eight patterns reported in the PDF-4/Organics 2013 database. Five of them are unindexed experimental patterns (PDF 00-057-1444, 00-058-1926, 00-059-1308, 00-062-1568, and 00-062-1569). The other three are calculated using the crystal structure data reported by Caira *et al.* (2004) and contained in the CSD. The first one (PDF 02-085-0756) corresponds to a phase containing one-quarter of an ethyl acetate molecule (Refcode IVUQEV), another (PDF 02-085-0757) to a monohydrated fluconazole (Refcode IVUQIZ), and the third (PDF 02-085-0758) to anhydrous fluconazole (Refcode IVUQOF).

As part of the work being carried out in our laboratory to explore the possible formation of polymorphs of important APIs under different crystallization conditions, high-quality powder diffraction data for anhydrous clotrimazole and monohydrated fluconazole were recorded and analyzed. The chemical nature of the materials under study was examined by FT-IR spectroscopy and thermogravimetric analysis/differential thermal analysis (TGA/DTA).

II. EXPERIMENTAL

A. Crystallization experiments

Clotrimazole and fluconazole, provided by Laboratorios CAM, C.A., were recrystallized by slow evaporation from different solvents. Several experiments were carried out in

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acetone, methanol, and methanol/H_2O (with a 4:1 volume ratio) at room temperature. Recrystallization of clotrimazole was also tried in glacial acetic acid. The materials obtained after each crystallization experiment were analyzed by IR spectroscopy, thermal analysis and X-ray powder diffraction.

B. IR spectroscopy and thermal analysis

FT-IR spectra were recorded in KBr pellets, using a Perkin Elmer RX1 spectrophotometer with Spectrum software. Thermogravimetric and derivative thermogravimetric analysis (TGA/DTG) and DTA were carried out in a SDT Q600V3 thermal analyzer using samples of 7–8 mg, heated up to 600 °C at a rate of 10 °C min^{-1}, under a dynamic nitrogen atmosphere at 100 ml min^{-1}.

C. X-ray powder diffraction data collection

Powder diffraction patterns were recorded at room temperature on a BRUKER D8 ADVANCE diffractometer working in the Bragg–Brentano geometry using CuKa radiation (Λ = 1.54184 Å), operating at 40 kV and 30 mA. The patterns were recorded in steps of 0.015 26° (2Θ), from 5 to 70° at 1.5 s $step^{-1}$. The diffractometer was equipped with primary and secondary Soller slits of 2.5°, divergence slit of 0.2 mm, Ni filter of 0.02 mm, and a LynxEye detector. The profile fit of each pattern was carried out with the FULLPROF software (Rodriguez-Carvajal, 1990). After the peak positions were established, the indexing of the patterns was performed with the program DICVOL06 (Boultif and Louër, 2004).

III. RESULTS AND DISCUSSION

The FT-IR spectra of clotrimazole recrystallized in all the solvents used were very similar and contain the expected vibrations associated with this molecule. The stretching vibration of the imidazole C–H appears at 3111.49 cm^{-1}, as expected. Aromatic C–H stretching vibrations appear at 3083.41, 3062.12, 3027.05, and 3005.49 cm^{-1}. The C = C stretchings in the aromatic rings are observed at 1617.81, 1586.78, 1565.56, and 1490.81 cm^{-1}. The absorption band of the C = N in the imidazole ring appears at 1347.38 cm^{-1} and the C–Cl stretch at 765.38 cm^{-1}. No absorption bands associated with presence of water or other solvent were observed. Similarly, the spectra registered for all of the recrystallized fluconazole samples were the same. The absorption observed at 3156.02 cm^{-1} is consistent with the presence of water. The absorption at 3107.85 cm^{-1} is associated with the C–H stretch of the triazole rings. The C–H stretches of the 2,4-difluorobenzyl group appear at 3062.46 and 3019.94 cm^{-1} and the C = C stretches of this group appear at 1619.65 and 1592.44 cm^{-1}. The absorptions at 1369.94 and 1248.99 cm^{-1} correspond to the C = N stretches of the triazole rings. The C–F and C–OH stretches appear at 1276.24 and 1018.70 cm^{-1}, respectively. The results obtained from the fluconazole IR spectra are consistent with those reported by Caira *et al.* (2004).

The TGA–DTG curves for raw and all recrystallized clotrimazole show the absence of solvent and no weight loss up to approximately 200 °C. Above this temperature, a series of decomposition processes take place in at least four steps up to 600 °C. The corresponding DTA trace of the methanol recrystallized material shows an endotherm at 147.71 °C, which corresponds to the melting point and agrees with the

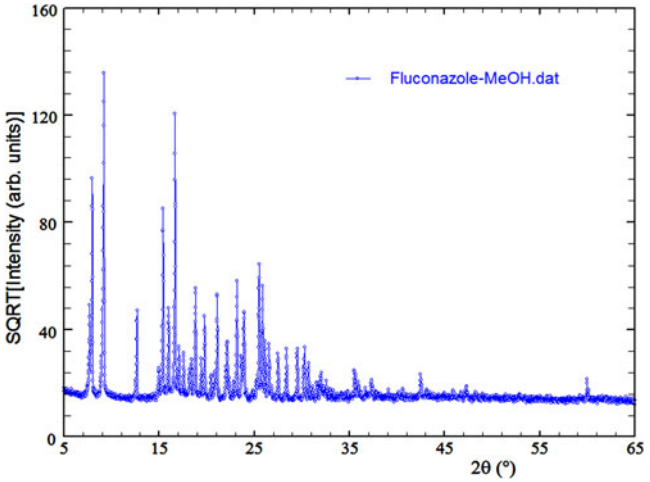

Figure 3. (Color online) Powder diffraction pattern of monohydrated fluconazole recrystallized in methanol.

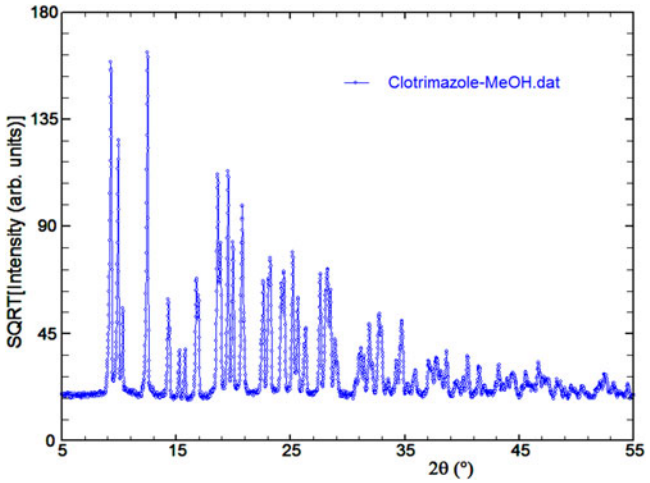

Figure 2. (Color online) Powder diffraction pattern of clotrimazole recrystallized in methanol.

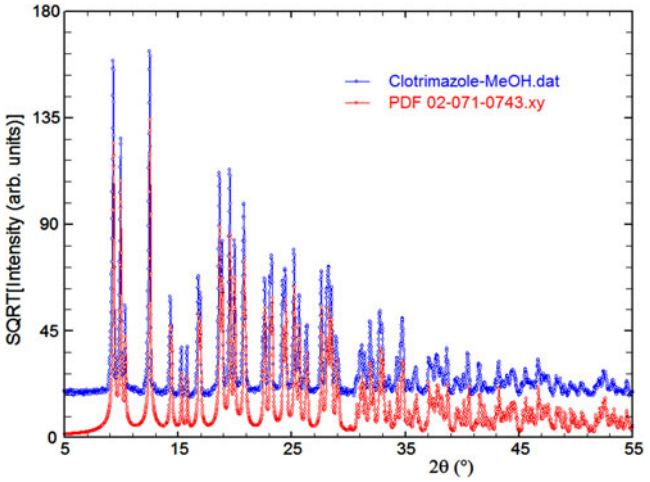

Figure 4. (Color online) Comparison of the powder diffraction pattern of clotrimazole recrystallized in methanol (blue) with the pattern calculated in PDF 02-071-0743 (red).

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TABLE I X-ray powder diffraction data of clotrimazole recrystallized in methanol.

No.	$2\theta_{obs}$(°)	d_{obs}(Å)	I_{obs}/I_o	h	k	l	$2\theta_{calc}$(°)	d_{calc}(Å)	$\Delta(2\theta)$
1	9.288	9.5215	952	0	0	1	9.292	9.5175	0.004
2	9.938	8.9001	596	0	−1	1	9.949	8.8903	0.011
3	10.340	8.5550	116	1	0	0	10.362	8.5367	0.022
4	12.496	7.0834	1000	−1	1	0	12.502	7.0800	0.006
5	14.315	6.1871	132	1	−1	1	14.321	6.1847	0.006
6	14.427	6.1394	83	−1	−1	1	14.447	6.1309	0.020
7	15.310	5.7932	53	1	1	0	15.294	5.7872	0.016
8	15.793	5.6113	55	0	1	1	15.801	5.6085	0.008
9	16.770	5.2865	170	−1	1	1	16.783	5.2826	0.013
10	16.957	5.2286	136	0	−2	1	16.969	5.2250	0.012
11	18.642	4.7597	470	0	0	2	18.646	4.7587	0.004
12	18.864	4.7041	259	1	−2	1	18.859	4.7054	−0.005
13	19.543	4.5422	478	−1	0	2	19.536	4.5439	−0.007
14	19.973	4.4454	260	0	−2	2	19.974	4.4452	0.001
15	20.789	4.2727	368	2	0	0	20.810	4.2684	0.021
16	22.631	3.9289	170	−1	−2	2	22.633	3.9286	0.002
17	23.097	3.8507	175	1	0	2	23.078	3.8539	−0.019
18	23.239	3.8275	221	1	2	0	23.256	3.8248	0.017
19	24.213	3.6757	164	−1	2	1	24.210	3.6762	−0.003
20	24.431	3.6434	190	2	0	1	24.438	3.6424	0.007
21	25.207	3.5330	235	−2	0	2	25.212	3.5322	0.005
22	25.662	3.4713	136	0	−3	1	25.659	3.4717	−0.003
23	26.341	3.3834	84	0	−3	2	26.344	3.3830	0.003
24	27.602	3.2316	186	1	−3	2	27.592	3.2328	−0.010
25	28.080	3.1777	143	−1	0	3	28.062	3.1797	−0.018
26	28.243	3.1597	196	2	−1	2	28.231	3.1611	−0.012
27	28.492	3.1326	150	−2	−2	1	28.490	3.1329	−0.002
28	28.908	3.0885	69	1	−1	3	28.899	3.0895	−0.009
29	30.737	2.9088	25	−2	−1	3	30.749	2.9076	0.012
30	31.039	2.8812	49	−3	0	1	31.026	2.8824	−0.013
31	31.166	2.8697	57	−1	2	2	31.179	2.8685	0.013
32	31.427	2.8465	47	3	0	0	31.437	2.8456	0.010
33	31.914	2.8041	90	0	2	2	31.913	2.8042	−0.001
34	32.166	2.7827	39	1	3	0	32.177	2.7818	0.011
35	32.768	2.7330	103	−1	1	3	32.763	2.7334	−0.005
36	32.952	2.7181	87	3	−1	1	32.966	2.7170	0.014
37	33.571	2.6694	25	0	3	1	33.577	2.6689	0.006
38	34.284	2.6155	43	−3	−1	2	34.289	2.6152	0.005
39	34.746	2.5818	95	2	−2	3	34.753	2.5813	0.007
40	35.307	2.5420	22	−3	1	2	35.309	2.5419	0.002
41	35.938	2.4988	33	1	2	2	35.949	2.4981	0.011
42	37.100	2.4232	42	−2	−3	3	37.082	2.4244	−0.018
43	37.787	2.3807	45	2	−4	1	37.780	2.3811	−0.007
44	38.138	2.3596	32	−3	3	0	38.131	2.3600	−0.007
45	38.650	2.3295	53	3	1	1	38.655	2.3292	0.005
46	39.537	2.2793	24	−3	2	2	39.548	2.2787	0.011
47	40.131	2.2469	26	3	2	0	40.118	2.2476	−0.013
48	40.500	2.2273	48	0	2	3	40.492	2.2277	−0.008
49	41.488	2.1765	37	−4	1	0	41.469	2.1774	−0.019
50	41.949	2.1536	20	−1	1	4	41.986	2.1518	0.037
51	42.997	2.1035	22	−4	0	2	42.989	2.1039	−0.008
52	43.227	2.0929	38	0	4	1	43.220	2.0932	−0.007
53	43.512	2.0798	20	−3	−1	4	43.516	2.0797	0.004
54	43.667	2.0728	21	3	−1	3	43.690	2.0718	0.023
55	43.982	2.0587	26	−4	2	1	43.980	2.0588	−0.002
56	44.329	2.0434	27	−3	−3	3	44.323	2.0436	−0.006
57	44.431	2.0389	30	1	3	2	44.402	2.0402	−0.029
58	44.527	2.0348	31	−3	−2	4	44.509	2.0356	−0.018
59	45.287	2.0024	20	4	0	1	45.294	2.0021	0.007
60	45.588	1.9898	32	−2	−2	5	45.581	1.9901	−0.007
61	46.705	1.9448	40	−4	−1	3	46.717	1.9444	0.012
62	47.176	1.9265	27	2	0	4	47.164	1.9269	−0.012
63	47.594	1.9105	25	−3	−3	4	47.583	1.9110	−0.011
64	48.298	1.8843	24	−4	3	1	48.294	1.8845	−0.004
65	48.596	1.8735	23	−2	5	0	48.595	1.8735	−0.001
66	48.995	1.8591	18	−4	−2	3	48.981	1.8596	−0.014

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TABLE I Continued

No.	$2\theta_{obs}$(°)	d_{obs}(Å)	I_{obs}/I_o	h	k	l	$2\theta_{calc}$(°)	d_{calc}(Å)	$\Delta(2\theta)$
67	50.615	1.8034	20	−3	−2	5	50.606	1.8037	−0.009
68	52.094	1.7556	23	−4	3	2	52.100	1.7554	0.006
69	52.473	1.7438	28	1	−5	5	52.475	1.7438	0.002
70	53.279	1.7193	22	4	−1	3	53.276	1.7194	−0.003
71	54.515	1.6832	21	4	2	1	54.518	1.6831	0.003

reported value of 147–149 °C, in the Merck Index (O'Neil *et al.*, 2006).

For the recrystallized fluconazole samples, the TGA–DTG traces exhibit a weight loss of about 5.18% indicating the presence of a water molecule. This water content may come from the hydrated raw material and/or from the presence of water in the methanol used. In the sample recrystallized in methanol, this dehydration occurs at 105.50 °C as indicated in the DTA by an endotherm. A second endotherm indicates that the melting of the dehydrated material takes place at 141.83 °C, in agreement with the value reported in the Merck Index (O'Neil *et al.*, 2006). The decomposition process begins at around 200 °C.

The powder diffraction patterns recorded for the samples recrystallized in acetone, methanol, and methanol/H_2O were very similar among them and similar to the pattern recorded for raw clotrimazole. The pattern of raw fluconazole contained the diffraction maxima of the patterns recorded for the recrystallized samples and four additional maxima, which coincide

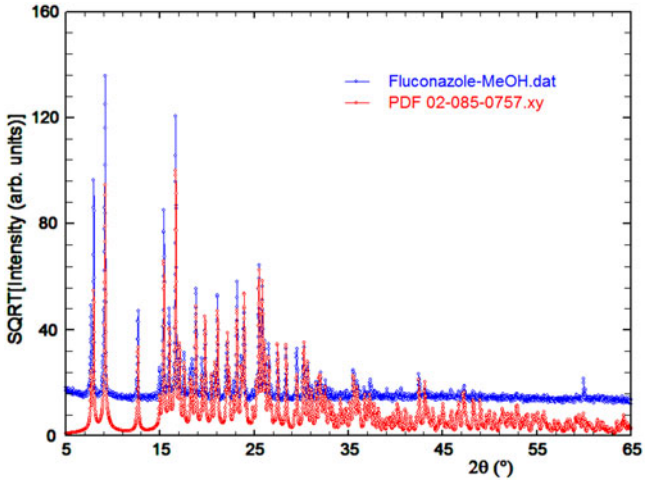

Figure 5. (Color online) Comparison of the powder diffraction pattern of monohydrated fluconazole recrystallized in methanol (blue) with the pattern calculated in PDF 02-085-0757 (red).

TABLE II X-ray powder diffraction data of fluconazole recrystallized in methanol.

No.	$2\theta_{obs}$(°)	d_{obs}(Å)	I_{obs}/I_o	h	k	l	$2\theta_{calc}$(°)	d_{calc}(Å)	$\Delta(2\theta)$
1	7.657	11.5456	134	0	0	1	7.665	11.5332	0.008
2	7.943	11.1305	504	0	1	0	7.954	11.1157	0.011
3	9.162	9.6521	1000	0	1	1	9.173	9.6403	0.011
4	12.647	6.9991	123	0	−1	1	12.662	6.9907	0.015
5	14.947	5.9269	35	0	1	2	14.946	5.9274	−0.001
6	15.383	5.7599	393	0	2	1	15.395	5.7554	0.012
7	15.972	5.5488	123	1	0	0	15.991	5.5423	0.019
8	16.653	5.3234	768	1	0	1	16.656	5.3224	0.003
9	17.069	5.1946	63	1	1	1	17.073	5.1933	0.004
10	17.552	5.0527	54	1	1	0	17.555	5.0518	0.003
11	18.195	4.8756	36	−1	1	0	18.205	4.8729	0.010
12	18.405	4.8204	46	0	2	2	18.406	4.8202	0.001
13	18.792	4.7220	173	−1	0	1	18.791	4.7221	−0.001
14	19.412	4.5726	48	0	−1	2	19.420	4.5707	0.008
15	19.763	4.4921	110	−1	1	1	19.762	4.4923	−0.001
16	20.449	4.3430	29	1	0	2	20.461	4.3404	0.012
17	20.845	4.2613	32	1	2	1	20.840	4.2624	−0.005
18	21.084	4.2136	161	−1	−1	1	21.084	4.2135	0.000
19	21.995	4.0411	35	0	1	3	21.989	4.0422	−0.006
20	22.131	4.0165	71	1	2	0	22.133	4.0161	0.002
21	22.332	3.9808	27	1	2	2	22.335	3.9803	0.003
22	22.811	3.8983	24	0	3	1	22.811	3.8983	0.000
23	23.141	3.8435	181	0	0	3	23.135	3.8444	−0.006
24	23.626	3.7657	43	−1	2	1	23.613	3.7677	−0.013
25	23.684	3.7566	50	0	2	3	23.695	3.7548	0.011
26	23.898	3.7234	120	−1	0	2	23.905	3.7223	0.007
27	24.172	3.6818	18	0	3	2	24.178	3.6809	0.006
28	24.859	3.5816	17	1	1	3	24.862	3.5812	0.003
29	25.233	3.5294	25	1	−2	1	25.237	3.5288	0.004
30	25.474	3.4965	228	0	−2	2	25.483	3.4954	0.009
31	25.825	3.4498	174	−1	−2	1	25.820	3.4505	−0.005

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TABLE II Continued

No.	$2\theta_{obs}$(°)	d_{obs}(Å)	I_{obs}/I_o	h	k	l	$2\theta_{calc}$(°)	d_{calc}(Å)	$\Delta(2\theta)$
32	26.113	3.4124	71	1	0	3	26.116	3.4120	0.003
33	26.525	3.3603	67	−1	−1	2	26.516	3.3614	−0.009
34	26.788	3.3279	21	0	−1	3	26.790	3.3276	0.002
35	27.472	3.2466	53	0	−3	1	27.478	3.2459	0.006
36	28.369	3.1460	60	1	3	0	28.363	3.1466	−0.006
37	29.493	3.0286	58	0	1	4	29.486	3.0292	−0.007
38	30.156	2.9635	30	0	2	4	30.154	2.9637	−0.002
39	30.252	2.9543	64	−1	0	3	30.246	2.9548	−0.006
40	30.659	2.9160	42	0	4	1	30.659	2.9160	0.000
41	31.078	2.8776	20	0	4	2	31.077	2.8777	−0.001
42	31.556	2.8351	23	1	2	4	31.553	2.8354	−0.003
43	31.913	2.8042	24	1	−3	1	31.903	2.8051	−0.010
44	31.997	2.7970	31	−1	−3	1	32.000	2.7968	0.003
45	32.298	2.7717	19	2	0	0	32.304	2.7712	0.006
46	32.531	2.7523	25	0	−3	2	32.533	2.7522	0.002
47	32.976	2.7162	17	2	1	0	32.956	2.7178	−0.020
48	33.690	2.6603	14	−2	1	0	33.683	2.6608	−0.007
49	34.140	2.6262	15	2	2	1	34.142	2.6261	0.002
50	34.416	2.6058	13	−2	0	1	34.417	2.6057	0.001
51	34.535	2.5971	15	2	2	2	34.541	2.5966	0.006
52	34.772	2.5799	16	1	−2	3	34.780	2.5793	0.008
53	35.507	2.5282	35	0	−4	1	35.503	2.5285	−0.004
54	35.600	2.5218	31	−2	−1	1	35.606	2.5214	0.006
55	35.912	2.5006	24	2	1	3	35.904	2.5011	−0.008
56	36.232	2.4792	15	1	−1	4	36.235	2.4791	0.003
57	36.693	2.4491	18	2	2	3	36.686	2.4496	−0.007
58	37.284	2.4117	25	−1	0	4	37.284	2.4117	0.000
59	37.747	2.3831	16	2	−2	1	37.753	2.3828	0.006
60	39.059	2.3061	17	0	3	5	39.057	2.3062	−0.002
61	40.163	2.2452	16	0	−4	2	40.163	2.2452	0.000
62	40.580	2.2231	18	0	5	0	40.579	2.2231	−0.001
63	42.460	2.1289	30	0	4	5	42.437	2.1300	−0.023
64	43.072	2.1001	17	−1	−3	3	43.062	2.1005	−0.010
65	43.406	2.0847	16	−1	5	1	43.420	2.0840	0.014
66	44.392	2.0406	14	−1	−2	4	44.396	2.0404	0.004
67	44.865	2.0202	14	0	2	6	44.844	2.0211	−0.021
68	44.976	2.0155	14	−2	−1	3	44.982	2.0152	0.006
69	45.864	1.9785	17	0	−4	3	45.855	1.9789	−0.009
70	45.971	1.9741	16	0	3	6	45.931	1.9758	−0.040
71	46.701	1.9450	15	−2	3	3	46.714	1.9445	0.013
72	47.165	1.9269	16	1	−5	1	47.155	1.9273	−0.010
73	47.271	1.9228	20	2	0	5	47.269	1.9229	−0.002
74	48.225	1.8870	14	0	−5	2	48.232	1.8867	0.007
75	48.966	1.8602	14	2	−4	1	48.949	1.8608	−0.017
76	55.769	1.6483	13	1	−2	6	55.771	1.6483	0.002
77	59.946	1.5431	26	0	−5	4	59.919	1.5437	−0.027
78	60.668	1.5264	12	1	−4	5	60.654	1.5267	−0.014
79	67.107	1.3948	10	4	2	2	67.105	1.3948	−0.002
80	67.792	1.3823	11	4	1	0	67.775	1.3826	−0.017

with some of the most intense peaks of the unindexed experimental pattern reported in entry PDF 00-059-1308. The X-ray powder patterns obtained for clotrimazole and fluconazole, recrystallized in methanol, are shown in Figures 2 and 3, respectively. In both patterns the square root of the intensity is plotted against 2θ in order to better show the weak diffraction maxima.

The diffraction patterns of recrystallized clotrimazole matched a pattern reported in the PDF-4/Organics (entry: PDF 02-071-0743) which corresponds to a pattern calculated using the crystal structure data reported in entry with refcode PUVRIH of the CSD for anhydrous clotrimazole (Figure 4). This is in accordance with the TGA/DTA results. The indexing of the best pattern recorded, from the sample recrystallized in methanol, carried out with the computer program DICVOL06 (Boultif and Louër, 2004), using the first 20 peaks, produced a triclinic unit cell. The analysis of all the 71 diffraction maxima registered, performed with NBS*AIDS83 (Mighell *et al.*, 1981), using the unit cell obtained by DICVOL06 led to the following unit cell parameters: $\boldsymbol{a} = 8.776(1)$ Å, $\boldsymbol{b} = 10.571(2)$ Å, $\boldsymbol{c} = 10.622(3)$ Å, $\alpha = 114.08(2)°$, $\beta = 96.87(2)°$, $\gamma = 97.61(2)°$, $V = 875.2(2)$ Å^3. The de Wolff (de Wolff, 1968) and Smith–Snyder (Smith and Snyder, 1979) figures of merit obtained were $M_{20} = 22.2$ and $F_{30} = 40.5(0.0098, 76)$, respectively. The results are presented in Table I. The diffraction pattern recorded for the sample recrystallized in glacial

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acetic acid was notably different from the other patterns registered. Single-crystal data are being collected in order to determine the structure of this potentially new phase.

In the case of fluconazole, the patterns of the recrystallized samples are very similar to the pattern reported in entry PDF 02-085-0757, calculated from entry IVUQIZ of the CSD, which corresponds to a monohydrated fluconazole. As it was the case for clotrimazole, for fluconazole, the sample obtained after recrystallization in methanol produced the best diffraction pattern. The comparison of this pattern with the calculated pattern of PDF 02-085-0757 is shown in Figure 5. The best result obtained in the indexing of the first 20 peaks of this pattern, carried out with DICVOL06 (Boultif and Louër, 2004), was a triclinic unit cell. The analysis, using this triclinic unit cell, carried out with NBS*AIDS83 (Mighell *et al.*, 1981) for the 80 diffraction maxima recorded, produced the following unit cell parameters: $\boldsymbol{a} = 5.6353(4)$ Å, $\boldsymbol{b} = 11.753(1)$ Å, $\boldsymbol{c} = 12.326(1)$ Å, $\alpha = 71.220(8)°$, $\beta = 79.896(9)°$, $\gamma = 84.35(1)°$, and $V = 760.13(9)$ Å^3. The de Wolff (de Wolff, 1968) and Smith–Snyder (Smith and Snyder, 1979) figures of merit obtained were $M_{20} = 48.2$ and $F_{30} = 117.8$ (0.0065, 39), respectively. The results of this analysis are presented in Table II. Both compounds crystallize in the triclinic system, with space group $P\bar{1}$, and $Z = 2$, as indicated in the Cambridge Structural Database.

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