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Determination of the Crystallographic Parameters of 2,2-Diphenyl-[1,3,2]Dithiagermole-4,5-Dicarbonitrile by Using X-Ray Powder Diffraction

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ABSTRACT

Using X-ray powder diffraction technique, diffraction pattern of compound of 2,2-diphenyl-[1,3,2]dithiagermole-4,5-dicarbonitrile was recorded. Profiting pattern, the crystal lattice and crystallographic parameters of the title compound were determined using analytic method and ITO computer program. According to the analytical method, the crystal system is monoclinic and unit cell parameters are; a=15.7202 Å, b=14.0053 Å, c=15.2543 Å, β=105.265º. Using ITO program, the type of crystal lattice was found as monoclinic and parameters of unit cell are a=15.7630 Å, b=14.0094 Å, c=15.3186 Å, β=105.451º. The results of the program are in agreement with those of analytical method.

Key Words: X-ray powder diffraction, 2,2-diphenyl-[1,3,2]dithiagermole-4,5-dicarbonitrile, Crystallographic parameters

1. INTRODUCTION

Unit cell parameters and crystal system of the title compound can be found with the help of X-ray diffraction pattern. Using the pattern, Bragg angles and the planar distances can be determined at first phase. Bragg angles and the planar distances are directly connected with Miller indices and unit cell parameters.

A multitude of methods have been designed to determine unit cell parameters and crystal system from the diffraction pattern of crystal structure. Some of these methods are in the content of computer programs while others are in the form of analytical procedures [1].

The main aim of this paper, using X-ray powder diffraction method, is to find the crystal system and unit cell parameters of the title compound whose structure was previously investigated with X-ray single crystal method [2], and to index its diffraction pattern. In this procedure, first the analytical method was used and then the ITO programme [3,4] included in WIN-INDEX program system [5].

2. EXPERIMENTAL

To determine the diffraction pattern of 2,2-diphenyl-[1,3,2]dithiagermole-4,5-dicarbonitrile compound, it was ground into powder in an agat mortar. Diffraction data was collected in a Bruker Axs D8 mark X-ray Powder Diffractometer, with a constant sweep velocity of 0.006°/s. In diffraction pattern, angle positions and relative intensities of Bragg reflections were determined. The diffraction pattern obtained from the mentioned sample is shown in Figure 1 and the data regarding the powder diffraction pattern is given in Table 1.

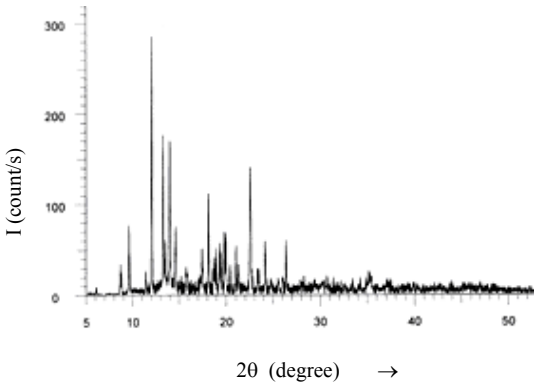

Figure 1. X-ray powder diffraction pattern of 2,2-diphenyl-[1,3,2]dithiagermole-4,5-dicarbonitrile compound.

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Using the data obtained from the powder diffraction pattern, in the analytic method and ITO computer program, the unit-cell parameters of the title compound and the crystal system were determined. Analytical method was based on mathematical operations formed using the distance of planes and Bragg equation. Data in this method were utilized cubic, tetragonal, hexagonal, rhombohedral, orthorhombic, monoclinic and triclinic test procedures, according to diminishing order of symmetry. The stages of the study done is explained below.

Cubic Test: The equation between the interplane distance and the unit cell edges is $d_{hkl} = \frac{a}{\sqrt{h^2+k^2+l^2}}$. When this equation is put to the Bragg law ($2d\sin\theta_{hkl} = \lambda$), then $\sin^2\theta_{hkl} = A(h^2 + k^2 + l^2)$ is obtained. A is $\frac{\lambda^2}{4a^2}$. For $(h^2 + k^2 + l^2)$, there are some permitted numerical values [6].

Table 1. The data related with the diffraction pattern of the compound.

2θ (°)	d (Å)	I (count/s)	% I
8.594	10.28119	23.4	8.6
9.548	9.25529	64.6	23.8
11.333	7.80141	20.4	7.5
12.001	7.36848	271	100.0
13.211	6.69648	157	57.7
13.432	6.58673	39.5	14.6
13.934	6.35043	148	54.6
14.535	6.08916	60.4	22.3
15.151	5.84294	18.0	6.6
17.383	5.09755	35.0	12.9
18.076	4.90354	99.4	36.7
18.525	4.78573	21.2	7.8
18.684	4.74525	24.1	8.9
18.859	4.70160	38.9	14.4
19.253	4.60647	43.5	16.0
19.406	4.57046	35.9	13.2
19.756	4.49012	56.1	20.7
19.887	4.46085	51.9	19.2
20.320	4.36680	27.0	10.0
20.990	4.22885	41.4	15.3
21.226	4.18251	24.9	9.2
22.484	3.95118	120	44.4
23.298	3.81503	20.7	7.6
24.074	3.69378	49.4	18.2
26.330	3.38207	51.1	18.8
23.390	3.80022	18.6	6.9

In this test, general factor A is found using the method of trial and error. So, the cell constant a is calculated. In order to understand whether 2,2-diphenyl-[1,3,2]dithiagermole-4,5 dicarbonitrile compound has a cubic structure or not, a table has been formed by dividing $\sin^2\theta$ by the permissible numbers [6]. Since a common numerical value did not occur in the table, it was concluded that the sample did not have a cubic crystal system.

Tetragonal Test: For tetragonal system, interplane distance was accepted as $d^2_{hkl} = \frac{1}{\frac{h^2+k^2}{a^2}+\frac{l^2}{c^2}}$ [6]. Put to the Bragg law, when $\frac{\lambda^2}{4a^2} = A$ and $\frac{\lambda^2}{4c^2} = C$ equations were utilized, $\sin^2\theta_{hkl} = A(h^2 + k^2) + Cl^2$ equation is obtained. In order to understand whether or not the sample to be studied is in tetragonal system, the procedure to be followed is to calculate A and C using the $\sin^2\theta$ observed and the values of integer number for *h, k* and *l*. For hk0 planes, where *l* is zero, the equation above is reduced to $\sin^2\theta_{hk0} = A(h^2 + k^2)$ equation. When the equation is solved, the ratio of the values of two $\sin^2\theta$ must be 2. When the table prepared with $\sin^2\theta$ value was searched, to understand whether 2,2-diphenyl-[1,3,2]dithiagermole-4,5-dicarbonitrile compound has tetragonal structure, a numerical value specified in the relevant test did not occur. Therefore, it was accepted that the sample possessed a non-tetragonal structure.

Hexagonal Test: For hexagonal system, the interplanar distances of planes are given by an equation of $d^2_{hkl} = \frac{1}{\frac{4(h^2+hk+k^2)}{3a^2}+\frac{l^2}{c^2}}$ [6]. If $\frac{\lambda^2}{3a^2} = A$ and , $\frac{\lambda^2}{4c^2} = C$ are accepted, the Bragg law is defined with $\sin^2\theta_{hkl} = A(h^2+hk+k^2)+Cl^2$ equation. If *l* index is zero, $\sin^2\theta_{hk0} = A(h^2 + hk + k^2)$ is obtained. When the ratio 3 occurs between the peaks, the probability of the symmetry to be hexagonal is high. It was accepted that the sample did not have a hexagonal structure. Since such a ration was not found in the ratio table, designed the sample 2,2-diphenyl-[1,3,2]dithiagermole-4,5-dicarbonitrile compound.

Rombohedral Test: In rombohedral system, $\frac{1}{d^2} = \frac{(h^2+k^2+l^2)\sin^2\alpha + 2(hk+kl+hl)(\cos^2\alpha-\cos\alpha)}{a^2(1-3\cos^2\alpha+2\cos^3\alpha)}$ equation represents the distance between planes [1]. While equations between Rombohedral miller indices (*p, q, r*) and Hexagonal miller indices (*h, k, l*) is expressed by *3p = h – k + 1, 3q = h + 2k + 1, 3r = - 2h - k + 1* the relationship between the rombohedral cell coefficients and the hexagonal cell coefficients is given by $a_2^2 = \frac{a^2}{3}+\frac{c^2}{9}$; $\sin\alpha/2 = \frac{3}{2}\cdot\frac{1}{\left[3+(c/a)^2\right]^{1/2}}$. Using these

equations for rombohedral system $\sin^2\theta_{pqr} = \frac{\lambda^2}{4}\left[\frac{\cos^2\alpha/2}{a^2\sin\alpha/2.\sin 3\alpha/2}\right]$ equation is obtained. As in hexagonal system, proportion must be sought. Following the procedure for 2,2-diphenyl-[1,3,2] dithiagermole-4,5-dicarbonitrile compound. We can see that the compound does not have a rombohedral system.

Orthorhombic Test: In orthorhombic system, the distance between the hkl planes is given by the equation $\frac{1}{d_{hke}} = \sqrt{\frac{h^2}{a^2}+\frac{k^2}{b^2}+\frac{l^2}{c^2}}$. If this equation is put to the Bragg law and solved using $\frac{\lambda^2}{4a^2} = A$, $\frac{\lambda^2}{4b^2}$ = B, $\frac{\lambda^2}{4a^2}$ = C the equation $\sin^2\theta_{hkl} = Ah^2 + Bk^2 + Cl^2$ is obtained. Some equations for $\sin^2\theta_{hkl}$ can be given to help indexing. Using these equations, $\sin^2\theta_{h00} = k^2$A, $\sin^2\theta_{0k0} = k^2$B, $\sin^2\theta_{00l} = k^2$C, $\sin^2\theta_{h00} + \sin^2\theta_{o\kappa o} = h^2$A + k^2B can be obtained, where $\sin^2\theta_{hk0} = h^2$A + k^2B. Hence, the below equation can be rewritten substituting equations above,

$$\sin^2\theta_{h_1k_10} = \sin^2\theta_{h_100} + \sin^2\theta_{0k_10}$$

$$\sin^2\theta_{h_1k_1l_1} = \sin^2\theta_{00l_1} + \sin^2\theta_{h_1k_10}$$

$$\sin^2\theta_{h_10l_1} = \sin^2\theta_{h_100} + \sin^2\theta_{00l_1}$$

by substracting the equations from each other,

$$\sin^2\theta_{0k_10} = \sin^2\theta_{h_1k_10} - \sin^2\theta_{h_100}$$

$$\sin^2\theta_{00l_1} = \sin^2\theta_{h_1k_1l_1} - \sin^2\theta_{h_1k_10}$$

$$\sin^2\theta_{00l_1} = \sin^2\theta_{h_2k_2l_1} - \sin^2\theta_{h_2k_20}$$

are obtained. The Hesse–Lipson method suggested to the index orthorhombic systems is based on these assumptions. Firstly using the observed $\sin^2\theta$ values, a table of differences is made in the Hesse–Lipson method. The procedure of determining the frequencies of differences was made and the difference value of the highest fruquency is accepted to correspond to one of the planes (100), (010), (001) and attempts are also made to index other peaks [6]. Since not all of the peaks have been indexed at the end of the procedure, we can say that our sample does not have an orthorhombic structure.

Monoclinic Test: For monoclinic system, the distance between planes is defined as $d_{hkl} = \frac{1}{\sqrt{\frac{h^2}{a^2}+\frac{1^2}{c^2}+\frac{2hl}{ac}\cos\beta}{\sin^2\beta}+\frac{k^2}{b^2}}}$ [1]. This equation has four unknowns. Thus, a subtance with monoclinic system can be indexed with greater difficulty than a subtances with high symmetry. The quadratic form for this system allows the establishment of some relationships considering the observed $\sin^2\theta$ values. The $\sin^2\theta$ values can be helpful in determining the two or three parameters and is used in the indexation of some of the peaks. The substitution of the interplane equation in the Bragg law and identification of certain parameters yield $\sin^2\theta_{hkl} = q_{hkl} = Ah^2 + Bk^2 + Cl^2 - Dhl$. The equations of the coefficients are as follows: $\frac{\lambda^2}{4a^2\sin^2\beta} = A$, $\frac{\lambda^2}{4b^2} = B$, $\frac{\lambda^2}{4c^2\sin^2\beta} = C$, $\frac{\lambda^2\cos^2\beta}{2ac\sin^2\beta} = D$. If we substitute the sinus expression for *hkl* plane, the equation $\sin^2\theta_{hkl}=q_{hkl}$ $=Ah^2+Bk^2+Cl^2+Dhl$ is found. If these equations are subtracted side by side, the equation $q_{hk\bar{l}} - q_{hkl} = 2Dhl$ is obtained. The next step is to make a table of differences to find the differences recurring at ratios such as 1:2:3:4:5. Using these ratios, we can calculate the differences $q_{hk\bar{l}} - q_{hkl} = 2Dhl$ and D value. When the table of differences made for the mentioned sample is studied (Table 2), the first, second, third, fourth folds and etc. of 0.0028 is found in the table of difference. Making use of the equation $q_{hk\bar{l}} - q_{hkl} = 2Dhl$, sample is tried to index. The equation $q_{10\bar{1}} - q_{101} = 2Dhl$ =0.0028 can be formed for k=0 plane. According to the equation, D values are equivalent 0.0014. If $q_{100} = A = 0.00561$ is accepted for A and the values are placed in the equation, $q_{101} = A + C - D$, C=0.00272 is obtained. With this value, the 4th peak is indexed with error 0.00005. Using $q_{202} = 4A + 4C - 4D$ equation, it is searched whether A, C and D values are equivalent whichever peaks and it is found that q_{202} = 0.02772 equation isn't equivalent whichever peak. Because the q_{303} and q_{404} values are also not found among the peaks, the assumption is incorrect. The operations are done again for the k = 1 planes. The D value is again found to be 0.0014 because of the $q_{11\bar{1}} - q_{111} = 2Dhl = 0.0028$. If $q_{110} = A + B = 0.00561$ is accepted, when the known values are written in their place in the $q_{11\bar{1}} = A + B + C + D$ equation, C is found to be 0.00274, using C values, $q_{002} = 4C = 0.01096$,

Table 2. The difference table of 2,2-diphenyl-[1,3,2]dithiagermole-4,5-dicarbonitrile compound.

No:	$\sin^2\theta$	1	2	3	4	5	6	7	8	9	10	11	12	13
1	0.00561													
2	0.00693	0.00132												
3	0.00975	0.00414	0.00282											
4	0.01093	0.00532	0.00400	0.00118										
5	0.01323	0.00762	0.00630	0.00348	0.00230									
6	0.01368	0.00807	0.00675	0.00393	0.00275	0.00045								
7	0.01471	0.00910	0.00778	0.00496	0.00378	0.00148	0.00103							
8	0.01600	0.01039	0.00907	0.00625	0.00507	0.00277	0.00232	0.00129						
9	0.01738	0.01177	0.01045	0.00763	0.00645	0.00415	0.00370	0.00267	0.00138					
10	0.02284	0.01723	0.01591	0.01309	0.01191	0.00961	0.00916	0.00813	0.00684	0.00546				
11	0.02468	0.01907	0.01775	0.01493	0.01375	0.01145	0.01100	0.00997	0.00868	0.00730	0.00184			
12	0.02591	0.02030	0.01898	0.01616	0.01498	0.01268	0.01223	0.01120	0.00991	0.00853	0.00307	0.00123		
13	0.02635	0.02074	0.01942	0.01660	0.01542	0.01312	0.01267	0.01164	0.01035	0.00897	0.00351	0.00167	0.00811	
14	0.02684	0.02123	0.01991	0.01709	0.01591	0.01361	0.01316	0.01213	0.01084	0.00946	0.00400	0.00216	0.00860	0.00049
15	0.02796	0.02235	0.02103	0.01821	0.01703	0.01473	0.01428	0.01325	0.01196	0.01058	0.00512	0.00328	0.00972	0.00161
16	0.02841	0.02280	0.02148	0.01866	0.01748	0.01518	0.01473	0.01370	0.01241	0.01103	0.00557	0.00373	0.01017	0.00206
17	0.02943	0.02382	0.02250	0.01968	0.01850	0.01620	0.01575	0.01472	0.01343	0.01205	0.00659	0.00475	0.01119	0.00308
18	0.02982	0.02421	0.02289	0.02007	0.01889	0.01659	0.01614	0.01511	0.01382	0.01244	0.00698	0.00514	0.01158	0.00347
19	0.03112	0.02551	0.02419	0.02137	0.02019	0.01789	0.01744	0.01641	0.01512	0.01374	0.00828	0.00644	0.01288	0.00477
20	0.03318	0.02757	0.02625	0.02343	0.02225	0.01995	0.01950	0.01847	0.01718	0.01580	0.01034	0.00850	0.01494	0.00683
21	0.03392	0.02831	0.02699	0.02417	0.02299	0.02069	0.02024	0.01921	0.01792	0.01654	0.01108	0.00924	0.01568	0.00757
22	0.03801	0.03240	0.03108	0.02826	0.02708	0.02478	0.02433	0.02330	0.02201	0.02063	0.01517	0.01333	0.01977	0.01166
23	0.04077	0.03516	0.03384	0.03102	0.02984	0.02754	0.02709	0.02606	0.02477	0.02339	0.01793	0.01609	0.02253	0.01442
24	0.04349	0.03788	0.03656	0.03374	0.03256	0.03026	0.02981	0.02878	0.02749	0.02611	0.02065	0.01881	0.02525	0.01714
25	0.05187	0.04626	0.04494	0.04212	0.04094	0.03864	0.03819	0.03716	0.03587	0.03449	0.02903	0.02719	0.03363	0.02552
26	0.04109	0.03548	0.03416	0.03134	0.03016	0.02786	0.02741	0.02638	0.02509	0.02371	0.01825	0.01641	0.02285	0.01474

Table 2 (continued). The difference table of 2,2-diphenyl-[1,3,2]dithiagermole-4,5-dicarbonitrile compound.

No:	$\sin^2\theta$	1	2	3	4	5	6	7	8	9	10	11	12	13
1	0.00561													
2	0.00693													
3	0.00975													
4	0.01093													
5	0.01323													
6	0.01368													
7	0.01471													
8	0.01600													
9	0.01738													
10	0.02284													
11	0.02468													
12	0.02591													
13	0.02635													
14	0.02684													
15	0.02796	0.00112												
16	0.02841	0.00157	0.00045											
17	0.02943	0.00259	0.00147	0.00102										
18	0.02982	0.00298	0.00186	0.00141	0.00039									
19	0.03112	0.00428	0.00316	0.00271	0.00169	0.00130								
20	0.03318	0.00634	0.00522	0.00477	0.00375	0.00336	0.00206							
21	0.03392	0.00708	0.00596	0.00551	0.00449	0.00410	0.00280	0.00074						
22	0.03801	0.01117	0.01005	0.00960	0.00858	0.00819	0.00689	0.00483	0.00409					
23	0.04077	0.01393	0.01281	0.01236	0.01134	0.01095	0.00965	0.00759	0.00685	0.00276				
24	0.04349	0.01665	0.01553	0.01508	0.01406	0.01367	0.01237	0.01031	0.00957	0.00548	0.00272			
25	0.05187	0.02503	0.02391	0.02346	0.02244	0.02205	0.02075	0.01869	0.01795	0.01386	0.01110	0.00838		
26	0.04109	0.01425	0.01313	0.01268	0.01166	0.01127	0.00997	0.00791	0.00717	0.00308	0.00032	-0.00240	-0.01078	

$q_{003} = 9C = 0.02466$ the 4th peak and the 11th peak are indexed with error 0.00003 and 0.0002, respectively. After this stage, the aim is to determine the unit cell parameters by finding the A and B values.

$q_{121} = A + 4B + C - D$,

$q_{120} = A + 4B$,

$q_{121} - q_{120} = C - D = 0.00274 - 0.0014 = 0.00134$

This difference value is between the 8th and 7th peaks. In this case, $q_{120} = 0.01471$ equation can be formed, A and B constants can be found by using the q_{120} and q_{110} reflections, $q_{120} = A + 4B = 0.01471$, $q_{110} = A + B = 0.00561$ after the operations made on the above equations A is found to be 0.00258 and B is found to be 0.00303 with these values,

$q_{112} = A + B + 4C - 2D = 0.00258 + 0.00303 + 0.01096 - 0.0028 = 0.01377$

$q_{122} = A + 4B + 4C - 2D = 0.00258 + 0.01212 + 0.01096 - 0.0028 = 0.02286$

$q_{12\bar{2}} = A + 4B + 4C + 2D = 0.00258 + 0.01212 + 0.01096 + 0.0028 = 0.02846$

$q_{130} = A + 9B + = 0.00258 + 0.02727 = 0.02985$

$q_{113} = A + B + 9C - 3D = 0.00258 + 0.00303 + 0.02466 - 0.0042 = 0.02607$

$q_{131} = A + 9B + C - D = 0.00258 + 0.02727 + 0.00274 - 0.0014 = 0.03119$

$q_{20\bar{2}} = 4A + 4C = 0.01032 + 0.01096 = 0.02118$

$q_{211} = 4A + B + C - 2D = 0.01032 + 0.00303 + 0.00274 - 0.0028 = 0.001329$

$q_{211} = 4A + B + C - 2D = 0.01032 + 0.00303 + 0.00274 - 0.0028 = 0.001329$

$q_{213} = 4A + B + 9C - 6D = 0.01032 + 0.00303 + 0.02466 - 0.0084 = 0.02961$

$q_{22\bar{1}} = 4A + 4B + C + 2D = 0.01032 + 0.01212 + 0.00274 + 0.0028 = 0.02798$

$q_{310} = 9A + B = 0.02322 + 0.00303 = 0.02625$

$q_{31\bar{1}} = 9A + B + C + 3D = 0.02322 + 0.00303 + 0.00274 + 0.0042 = 0.03319$

with the choices all of the peaks are indexed except for six peaks. The compound of 2,2-diphenyl-[1,3,2]dithiagermole-4,5-dicarbonitrile can be said to have a monoclinic system.

After the indexed process, the unit cell parameters are determined. To find the b value, if $B = \frac{\lambda^2}{4b^2}$ is used, b is found to be 14.0053 Å. If the necessary mathematical arrangements are done in expressions of A, C and D, the equation of $\cos\beta = \frac{D}{2\sqrt{AC}}$ is found. In the equation, when the D, A and C values are put in, their place, β=74.735°. There would not be change in the system if we take 105.265º as the β angle which completes the β value to 180º. From the A expression, $a = \frac{\lambda}{2\sin\beta\sqrt{A}}$ equation is found. Here, if β and A values are put into their places, $a = 15.7202$ Å is obtained and if similar operations are done on C, $c = 15.2543$ Å.

The results of the indexed process that we made for the compound of 2,2-diphenyl-[1,3,2]dithiagermole-4,5-dicarbonitrile are shown in Table 3.

Table 3. Analytical Result Table of 2,2-Diphenyl-[1,3,2]dithiagermole-4,5-dicarbonitrile compound.

h	k	l	$\sin^2\theta_{clc}$	$\sin^2\theta_{obs}$	$\Delta\sin^2\theta$
1	1	0	0.00561	0.00561	0.00000
1	1	1	0.00695	0.00693	0.00002
1	1	-1	0.00975	0.00975	0.00000
0	0	2	0.01096	0.01093	0.00003
2	1	1	0.01032	0.01032	0.00006
1	1	2	0.01377	0.00139	0.00009
1	2	0	0.01471	0.01471	0.00000
1	2	1	0.01600	0.01600	0.00000
1	2	2	0.02286	0.02284	0.00002
0	0	3	0.02466	0.02468	0.00002
1	1	3	0.02607	0.02591	0.00016
3	1	0	0.02625	0.02635	0.00010
2	0	2	0.02688	0.02684	0.00004
2	2	1	0.02798	0.02796	0.00002
1	1	2	0.02286	0.02841	0.00555
2	1	3	0.02961	0.02943	0.00018
1	3	0	0.02985	0.02982	0.00003
1	3	1	0.03119	0.03112	0.00007
3	1	-1	0.03319	0.03318	0.00001
				0.03392	
				0.03801	
				0.04077	
				0.04349	
				0.04100	

In the second part of the study, the unit cell parameters and crystal systems of the sample by using the ITO programme, which the WIN-INDEX program loaded in the system consists of, was evaluate by using the X-ray diffraction pattern of 2,2-diphenyl-[1,3,2]dithiagermole-4,5-dicarbonitrile compound. The use of ITO computer program for low symmetry systems is recommended by programmers. The peak number should be 20 least, the reason for this limitation is that the programme uses the first 20 lines during indexing.

By using the ITO programme, the three solutions were found for the mentioned sample. Among these, the first solution which has the highest Merit number and the lowest non-indexed peak number was taken as the reference. In Table 4, the solutions were given that the ITO programme found for 2,2-diphenyl-[1,3,2]dithiagermole-4,5-dicarbonitrile compound.

In Table 5, the results of selected solution were listed which was given the programme. If checked carefully, one can see on the table that there are some peaks, yet not calculated by the programme. The reason for this is that there might be some impurities in the structure of the sample. Mentioned impurities is determined as thiazyl boron fluoride amide sulfide (PDF file no. 84-0803) resulting from the former studied in agat mortor. In the table there are also peaks which are calculated by the programme, but not observed. This can be explained as the intensities of X-rays reflecting from some planes might be low. Because, it is really difficult to distinguish the peaks from the background, which are formed by the interference of such kind of X-ra

Table 4. The solutions were given that the ITO programme found for 2,2-diphenyl-[1,3,2]dithiagermole-4,5-dicarbonitrile compound.

Metric :

	Nr	a	b	c	Alpha	Beta	Gamma	Volume
*	1	15.7630	14.0094	15.3186	90.000	105.451	90.000	3260.545
	2	18.9625	15.8555	14.6884	90.000	105.827	90.000	4248.797
	3	23.8943	29.0146	6.8510	90.000	90.000	90.000	4749.689

Lattice and Quality Figures :

	Nr	Crystal System	Lattice Type	Not indexed	Fom	Single indexed	Zero shift
*	1	MONOC	P	1	22.93	14	0.0146
	2	MONOC	P	2	6.00	13	0.1453
	3	ORTHO	P	5	6.24	13	0.0360

Table 5. The results of ITO program for 2,2-difenil-[1,3,2]dithiagermol-4,5-dicarbonitril compound.

Nr	*h*	*k*	*l*	$2\theta_{obs}(°)$	$2\theta_{clc}(°)$	$\Delta\theta(°)$	d_{obs} (Å)	d_{clc}(Å)	Δd(Å)	I/I_o
1	*1*	*1*	*0*	8.594	8.593	0.000	10.2812	10.2818	-0.0006	8.6
2	*1*	*1*	*-1*	9.548	9.549	-0.000	9.2553	9.2549	0.0004	23.8
3	*1*	*1*	*1*	11.333	11.334	-0.000	7.8011	7.8011	0.0003	7.5
4	*0*	*0*	*2*	12.001	11.993	0.008	7.3685	7.3734	-0.0049	100.0
5	*2*	*1*	*-1*	13.211	13.214	-0.003	6.6965	6.6951	0.0014	57.7
6	*2*	*1*	*0*		13.262	-0.066		6.6707	0.0332	
7	*1*	*1*	*-2*	13.432	13.440	-0.008	6.5867	6.5830	0.0037	14.6
8	*1*	*2*	*0*	13.934	13.925	0.009	6.3504	6.3545	-0.0041	54.6
9	*1*	*2*	*-1*	14.535	14.538	-0.002	6.0892	6.0882	0.0010	22.3
10				15.151			5.8429			6.6
11	*1*	*2*	*-2*	17.383	17.358	0.025	5.0976	5.1047	-0.0072	12.9
12					17.399	-0.031		5.0928	0.0090	
13	*0*	*0*	*3*	18.076	18.024	0.052	4.0935	4.9176	-0.0141	36.7
14	*3*	*1*	*-1*		18.081	-0.020		4.9022	0.0053	
15	*1*	*1*	*-3*	18.525	18.523	0.002	4.7857	4.7861	-0.0004	7.8
16	*3*	*1*	*0*	18.684	18.630	0.055	4.7452	4.7591	-0.0138	8.9
17	*2*	*0*	*3*		18.689	-0.019		4.7442	0.0048	
18	*2*	*0*	*2*	18.859	18.861	-0.002	4.7016	4.7011	0.0005	14.4
19	*2*	*2*	*1*	19.253	19.251	0.002	4.6065	4.6069	-0.0004	16.0
20	*1*	*2*	*2*	19.406	19.408	-0.002	4.5705	4.56099	0.0006	13.2
21	*2*	*1*	*-3*	19.756	19.742	0.014	4.4901	4.4933	-0.0032	20.7
22	*1*	*3*	*0*	19.887	19.889	-0.002	4.4608	4.4604	0.0004	19.2
23	*2*	*1*	*2*		19.906	-0.033		4.4567	0.0074	
24	*0*	*3*	*1*		19.440	-0.067		4.4492	0.0149	
25	*1*	*3*	*-1*	20.320	20.327	-0.007	4.3668	4.3654	0.0015	10.0
26	*3*	*1*	*1*	20.990	20.982	0.008	4.2289	4.2305	-0.0017	15.3
27				21.226			4.1825			9.2
28				22.484			3.9512			44.4
29				23.298			3.8150			7.6
30				23.390			3.8002			6.9
31				24.074			3.6938			18.2
32				26.330			3.3821			18.8

3. RESULTS AND DISCUSSION

The unit cell parameters and the crystal system were found via the analytical method and ITO computer programme, by using data obtained from X-ray powder diffraction method of 2,2-diphenyl-[1,3,2]dithiagermole-4,5-dicarbonitrile compound. With the analytical method, the crystal system was found as monoclinic and unit cell parameter values were calculated as a = 15.7202 Å, b = 14.0053 Å, c = 15.2543 Å, β = 105.265°; with the ITO computer programme, a = 15.7630 Å, b = 14.0094 Å, c = 15.3186 Å, β = 105.451° were found for the monoclinic system. The single crystal data of the compound are a = 15.3121(11) Å, b = 14.0064(10) Å, c = 15.7730(11) Å, β = 105.3900(10) º and the crystal system is monoclinic system [2]. Thus, the results obtained in this work for structure investigations using X-ray powder diffraction are very satisfactory.

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