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CRYSTAL DATA FOR THREE SCHIFF BASE COMPLEXES

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INTRODUCTION

This study is a part of a general work which aims to investigate the complexes made by Schiff bases and transition metals (Maggio, Pizzino and Romano, 1974). The complexes between Schiff bases and transition metals have a great importance in coordination chemistry (Elerman, Paulus and Fuess, 1991). The aims of the present work are indexing of powder diffraction data, obtained from three Schiff bases, and precise lattice parameter determinations for possible crystal structural works. For this purpose, diffraction data of $C_{14}H_{13}ON$, $C_{21}H_{22}N_2O_2Ni$, $C_{17}H_{18}O_2N_2$ were obtained and lattice parameters were refined by applying least-squares method.

EXPERIMENTAL PROCEDURES

Origin of specimens

All compounds were synthesised, in Chemistry Department of Ankara University, Science Faculty, in the following way.

1° $C_{14}H_{13}ON$: 0.1 mol (12.2 gr) Salicylaldehyde is dissolved in 100 ml ethanol by heating and stirring in a magnetic stirrer. This solution is heated up to 60 °C. Then 0.1 mol benzylamine is added to it slowly. When this solution is left for cool down, yellow coloured prismatic crystals were precipitated (Atakol, 1986).

2° $C_{21}H_{22}N_2O_2Ni$: 0.005 mol N- (2-hydroxyphenyl)-2 Hydroxy-1 naphthaldimine is dissolved and heated up to its melting point in 250 ml methanol by heating and 2.0 ml diethylamine is added. 0.005 mol Ni $(CH_3COO)_2 4H_2O$ is dropwise dissolved in 50 ml hot ethanol. The mixture of the two solutions was then refluxed for three hours. Red crystals were formed during the reflux operation (Atakol, 1986).

3° $C_{17}H_{18}O_2N_2$: 0.2 mol salicylaldehyde is dissolved in 100 ml ethanol and heated up to 60 °C. Then solution of 1 mol (1.3) diaminoprophane in 20 ml ethanol is added. Yellow crystals were formed during the reflux operation (Atakol, 1986).

Data Collection

Diffraction data were collected with a Rigaku Powder diffractometer using a fine-focus Cu tube operated at 35 kV and 15 mA. Observable reflections were in the scanning range from 2° to 60°. A rateameter setting of 500 c / sec with a 2 sec time constant, the scan speed of 2° / min were used to detect the weak peaks. The data were obtained at room temperature i.e. approximately 23 °C.

Data reduction and Results

Preliminary crystal parameters and space groups for these complexes were obtained from zero and upper layer Weissenberg photographs. Reflections obtained by powder diffraction were indexed and then used in several cycles of unit cell parameter refinement, employing (Elmali and Kabak, 1989) the least-squares unit cell parameters refinement program. The results are given in Table 1. The powder diffraction patterns of three Schiff base complexes are given in Table 2. The calculated d values are in good agreement with the observed values of three compounds.

Table 1. Crystal data for three Schiff base complexes

1° $C_{14}H_{13}ON$	
a = 5.922 (2) Å	Space Group = $P2_1/C$
b = 13.381 (9) Å	Z = 4
c = 14.536 (8) Å	
β = 98.34 (3)°	
D_x = 1.231 gr / cm³	
V = 1139.2 Å³	
F_{30} = 5.931 (0.0809,62)	
2° $C_{21}H_{22}N_2O_2Ni$	
a = 12.231 (4) Å	Space Group = $P2_1/N$
b = 8.844 (2) Å	Z = 4
c = 17.156 (5) Å	
β = 96.04 (2)°	
D_x = 1.412 gr / cm³	
V = 1845.5 Å³	
F_{30} = 7.831 (0.0515,74)	
3° $C_{17}H_{18}O_2N_2$	
a = 13.448 (4) Å	Space Group = $P2_1/C$
b = 9.110 (3) Å	Z = 4
c = 13.883 (5) Å	
β = 117.33g	
D_x = 1.240 gr / cm³	
V = 1510.9 Å³	
F_{30} = 22.075 (0.325,42)	
F_N = (1 / |Δ2θ|) (N / N_{poss})	

Table 2. X-Ray powder diffraction data for three Schiff base complexes

1. $C_{14}H_{13}ON$

h	k	l	d_{obs}	d_{calc}	I/I_0	$(2\theta)_{exp}$	$(\Delta 2\theta)$
0	1	1	9.584	9.796	8	9.220	.200
0	0	−2	7.190	7.190	15	12.300	.001
0	2	0	6.681	6.690	20	13.240	.018
0	−1	−2	6.311	6.333	2	14.020	.050
0	2	−1	6.079	6.066	1	14.560	−.030
0	2	1	5.964	6.066	3	14.840	.250
1	−1	0	5.329	5.367	33	16.620	.117
1	1	0	5.317	5.367	33	16.660	.157
1	1	1	4.839	4.822	57	18.320	−.065
0	0	3	4.766	4.794	12	18.600	.107
1	−2	0	4.410	4.408	17	20.120	−.009
1	2	−1	4.304	4.350	24	20.620	.221
1	0	2	4.227	4.249	97	21.000	.111
1	−1	2	4.051	4.050	33	21.920	−.010
1	−2	−2	3.990	3.956	2	22.260	−.196
0	−2	3	3.911	3.897	39	22.720	−.082
0	−2	−3	3.884	3.897	75	22.880	.078
1	1	−3	3.857	3.839	100	23.040	−.111
1	−1	−3	3.814	3.839	55	23.300	.149
0	3	2	3.745	3.790	57	23.740	.289
0	0	−4	3.637	3.595	3	24.520	−.223
1	−3	0	3.548	3.548	9	25.080	.009
1	2	3	3.448	3.438	38	25.820	−.076
0	−4	0	3.346	3.345	16	26.620	−.005
0	−4	2	3.023	3.033	7	29.520	.096
0	4	2	3.011	3.033	10	29.640	.216
1	2	−4	2.959	2.949	16	30.180	−.105
1	4	0	2.899	2.905	7	30.820	.068
2	1	1	2.725	2.733	2	32.840	.101
2	2	−1	2.700	2.704	1	33.160	.056
2	2	1	2.573	2.577	2	34.840	.052
0	0	−6	2.390	2.397	5	37.600	.108
0	1	−6	2.362	2.360	2	38.060	−.052
0	1	6	2.354	2.359	2	38.200	.089
2	−1	3	2.324	2.316	6	38.720	−.127
2	2	−4	2.301	2.303	6	39.120	.037
1	5	2	2.262	2.264	2	39.820	.047
0	−2	−6	2.250	2.256	2	40.040	.119
2	3	2	2.235	2.236	3	40.320	.023
1	5	−3	2.225	2.225	5	40.500	.000
2	−4	0	2.206	2.204	1	40.880	−.035
1	2	6	2.013	2.015	2	45.000	.051
2	3	−5	1.994	1.988	1	45.440	−.160
3	1	−2	1.937	1.938	6	48.860	.010
1	−3	6	1.911	1.910	9	47.540	−.037
2	5	−3	1.891	1.893	2	48.080	.071
0	3	−7	1.867	1.866	2	48.740	−.021
1	1	7	1.838	1.838	5	49.540	.001
3	0	−4	1.832	1.832	5	49.740	.004
2	3	−6	1.828	1.827	3	49.840	−.013
3	1	2	1.798	1.802	3	50.740	.135
1	−5	−6	1.760	1.762	1	51.900	.056
1	6	−5	1.732	1.731	1	52.820	−.030
3	1	−5	1.723	1.723	6	53.100	−.005
3	3	2	1.683	1.684	1	54.480	.047

h	k	l	d_{obs}	d_{calc}	I/I_0	$(2\theta)_{exp}$	$\Delta(2\theta)$
3	–2	3	1.673	1.669	2	54.820	–.165
1	–5	6	1.660	1.658	1	55.280	–.070
2	–6	3	1.618	1.618	1	56.840	–.008
3	3	3	1.607	1.607	1	57.300	.028
3	4	2	1.595	1.598	1	57.740	.101
3	–5	–4	1.515	1.512	1	61.140	–.134
4	1	–1	1.470	1.470	1	63.180	–.017

2° $C_{21}H_{22}N_2O_3Ni$

h	k	l	d_{obs}	d_{calc}	I/I_0	$(2\theta)_{exp}$	$(\Delta 2\theta)$
1	0	–1	10.202	10.437	100	8.660	.195
1	0	1	9.243	9.445	3	9.560	.205
0	–1	0	8.981	8.844	1	9.840	–.153
0	0	2	8.418	8.530	7	10.500	.139
–1	1	1	6.712	6.747	9	13.180	.069
–1	–1	1	6.641	6.747	4	13.320	.210
2	0	0	6.021	6.082	28	14.700	.148
–1	0	3	5.407	5.373	1	16.380	–.104
1	1	2	5.279	5.321	6	16.780	.134
2	1	0	4.984	5.011	5	17.780	.096
–2	1	1	4.929	4.925	5	17.980	–.016
2	0	2	4.726	4.723	1	17.760	–.014
–1	1	3	4.581	4.592	20	19.360	.047
–2	–1	2	4.462	4.494	14	19.880	.143
1	–1	3	4.333	4.323	3	20.480	–.047
0	2	1	4.275	4.280	10	20.760	.026
–1	–2	0	4.149	4.156	1	21.400	.037
–1	–2	1	4.062	4.072	4	21.860	.050
0	2	2	3.914	3.926	13	22.700	.070
3	1	1	3.523	3.533	1	25.260	.078
2	–2	1	3.461	3.458	16	25.720	–.023
–2	–1	4	3.398	3.397	14	26.200	–.015
–2	2	3	3.106	3.116	7	28.720	.094
1	–1	5	3.013	3.008	2	29.620	–.052
3	–2	0	2.990	2.988	1	29.860	–.013
3	–2	1	2.976	2.983	1	30.000	.073
0	–3	0	2.951	2.948	1	30.260	–.033
1	–2	4	2.972	2.924	1	30.520	–.022
4	1	0	2.871	2.876	2	31.120	.046
–2	2	4	2.822	2.828	2	31.680	.070
–4	1	2	2.812	2.811	2	31.800	–.008
–1	–3	2	2.741	2.736	1	32.640	–.058
–3	–2	3	2.730	2.734	1	32.780	.054
2	3	1	2.599	2.603	7	34.480	.057
–4	2	1	2.512	2.510	1	35.720	–.016
2	0	6	2.474	2.478	1	36.280	.057
–5	1	2	2.325	2.323	3	38.700	–.036
–4	1	5	2.318	2.315	2	38.820	–.043
3	3	2	2.260	2.260	2	39.860	.012
–4	–2	4	2.244	2.247	3	40.160	.068
0	4	1	2.188	2.193	1	41.220	.086
3	–2	5	2.167	2.166	1	41.640	–.015
4	3	0	2.116	2.117	1	42.700	.018
–4	–2	5	2.104	2.109	2	42.940	.090

h	k	l	d_{obs}	d_{calc}	I/I_o	$(2\theta)_{exp}$	$(\Delta 2\theta)$
4	−2	4	2.083	2.083	4	43.400	−.006
1	3	6	1.990	1.994	1	45.540	.084
3	1	7	1.947	1.949	1	46.600	.042
−3	4	0	1.943	1.941	1	46.720	−.039
0	2	8	1.920	1.921	1	47.300	.019
6	1	2	1.883	1.883	2	48.300	.009
0	3	7	1.878	1.878	1	48.440	.023
1	2	8	1.870	1.870	1	48.640	−.002
4	−3	5	1.860	1.861	1	48.940	.033
−4	−2	7	1.829	1.828	1	49.820	−.020
3	−2	7	1.823	1.821	1	50.000	−.051
1	−4	6	1.713	1.712	1	53.440	−.029
−2	−4	6	1.708	1.707	2	53.620	−.020
−5	−3	5	1.703	1.704	2	53.780	.016
0	5	3	1.689	1.689	1	54.260	−.006
6	3	0	1.670	1.670	1	54.920	.000
4	0	8	1.665	1.666	1	55.120	.028
3	3	7	1.653	1.654	1	55.560	.049
4	3	1	1.647	1.649	1	55.764	.053
6	3	2	1.613	1.613	1	57.060	.014
−2	−5	4	1.595	1.594	1	57.740	−.050
−5	−2	8	1.583	1.582	1	58.220	−.043
−3	4	7	1.558	1.557	1	59.280	−.017
6	1	6	1.550	1.550	1	59.580	−.022
−3	−5	4	1.537	1.537	1	60.160	.010
−4	−5	1	1.523	1.530	1	60.460	.006
5	−1	8	1.505	1.504	1	61.560	−.063
−3	5	5	1.489	1.489	1	62.300	−.012
6	−4	1	1.479	1.479	1	62.780	−.012
−6	4	3	1.475	1.474	1	62.960	−.073

3° $C_{17}H_{18}O_2N_2$

h	k	l	d_{obs}	d_{calc}	I/I_o	$(2\theta)_{exp}$	$(\Delta 2\theta)$
1	1	7	7.196	7.180	14	12.300	−.027
−1	0	2	6.916	6.927	16	12.800	.022
2	0	0	5.961	5.973	7	14.860	.030
−2	0	2	5.806	5.833	16	15.260	.070
−1	1	2	5.478	5.514	29	16.180	.107
−2	−1	1	5.385	5.408	36	16.460	.071
2	1	0	4.983	4.995	53	17.800	.045
−2	−1	2	4.890	4.912	24	18.140	.082
0	−2	0	4.552	4.555	12	19.500	.012
1	−2	0	4.258	4.256	26	20.860	−.011
3	0	0	3.976	3.982	100	22.360	.036
−2	−2	1	3.770	3.770	36	23.600	.006
−2	−2	0	3.610	3.622	49	24.660	.083
−4	0	2	3.363	3.361	17	26.500	−.023
2	−2	1	3.248	3.240	7	27.460	−.069
3	−1	1	3.158	3.156	24	28.260	−.014
−4	−1	1	3.073	3.075	15	29.060	.016
0	2	3	3.046	3.052	3	29.320	.058
3	2	0	2.996	2.998	3	29.820	.020
−1	3	0	2.944	2.943	7	30.360	−.010

h	k	l	d_{obs}	d_{calc}	I/I_0	$(2\theta)_{exp}$	$(\Delta 2\theta)$
0	–1	4	2.935	2.921	12	30.460	–.149
4	–1	0	2.840	2.838	3	31.500	–.023
2	1	3	2.703	2.705	4	33.140	.028
–3	2	4	2.655	2.657	2	33.760	.027
–3	–1	5	2.617	2.617	2	34.260	.001
1	–1	5	2.564	2.563	5	35.000	–.010
–3	–3	2	2.495	2.497	3	36.000	.032
0	–3	3	2.445	2.443	1	36.760	–.035
–3	3	3	2.395	2.393	1	37.560	–.021
–5	–2	2	2.309	2.310	1	39.000	.017
–5	1	5	2.260	2.260	2	39.880	–.006
–1	4	0	2.238	2.237	3	40.300	–.013
–6	1	2	2.153	2.153	3	41.960	–.009
–6	–1	4	2.133	2.133	2	42.380	–.005
3	2	3	2.100	2.101	3	43.080	.032
2	–2	4	2.080	2.079	6	43.500	–.020
–4	–2	6	2.010	2.011	4	45.100	.007
5	0	2	1.947	1.947	3	46.660	.013
–5	2	6	1.913	1.914	2	47.520	.009
–5	3	0	1.878	1.878	2	48.460	–.018
–6	3	3	1.804	1.803	3	50.600	–.031
3	4	2	1.768	1.769	3	51.700	.031

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CRYSTAL DATA FOR $SrTeO_4$

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INTRODUCTION:

This study is a part of a general work which aims to investigate the some strontium tellurates. The aim of the present work is indexing of the powder diffraction data. The measured precise lattice parameters are also used in crystal structure determinations. For this purpose, precise lattice parameters were found by indexing powder diffraction data.

EXPERIMENTAL PROCEDURES

Origin of specimens:

$SrTeO_4$ was prepared by the solid state reactions between $SrCO_3$ and TeO_2. To obtain the substance following experiments was made under 3000 atmosphere pressure. The results of these experiments are given in Table 1.

Table 1. The results of the solid state reactions

$SrCo_3$:TeO_2	Reaction conditions	Identificated phase
45 : 55	650°C, 50 hour	$SrTeO_4$, $Sr_2Te_3O_8$
47.5 : 52.5	700°C, 50 hour	$SrTeO_4$, $Sr_2Te_3O_8$
49 : 51	700°C, 50 hour	$SrTeO_4$
50 : 50	700°C, 50 hour	$SrTeO_4$
50 : 50	825°C, 50 hour	$SrTeO_3$
51 : 49	700°C, 50 hour	$SrTeO_4$
55 : 45	900°C, 50 hour	$SrTeO_3$, $SrTeO_6$

We have obtained $SrTeO_4$ according to the following chemical reaction:

$$SrCO_3 + TeO_2 + 1/2\ O_2 \rightarrow SrTeO_4 + CO_2$$

According to this equation, tellurium was oxidated by the air oxygen from Te^{+4} to Te^{+6}

Data Collection:

Powder diffraction data for $SrTeO_4$ were recorded with a Guinier-Hägg, XDC-700 focusing camera with a casette radius of 50 mm using MoKα ($\lambda = 0.7107$ Å) radiation, Si powder was used as an internal calibration standard. Relative intensities were measured on photographic film with a Joyce-Loebl double-beam microdensitometer. The data were obtained at room temperature i.e. approximately 23 °C.

Data reduction:

Crystal data for $SrTeO_4$ were obtained from zero and upper layers Weissenberg photographs. The results are given in Table 1. Indexed reflections were used in several cycles of unit cell parameter refinement using the Elmalı and Kabak (1979) least-squares unit cell parameter refinement programme. The powder diffraction pattern of $SrTeO_4$ is given in Table 2. The calculated d values are in good agreement with the observed values.

Results and discussion:

After we obtained the $SrTeO_4$, DTA and TG analyses with the pure powder sample were made at up to 850 °C. After the heating from 720 °C to 850 °C an endothermal effect was observed.

X-Ray powder diffraction pattern before the DTA and TG analyses, consist only of the reflections belonging to $SrTeO_4$. Powder diffraction pattern after the DTA and TG analyses, showed the characteristic diagram of $SrTeO_3$. During these DTA and TG analyses a loss of mass was observed with the value of 5.8 %. This value is in good agreement with the value of 5.7 % calculated for the decomposition of $SrTeO_4$ to $SrTeO_3$. Such experiments were repeated under normal atmospheric pressure in an oven. Pure powder sample of $SrTeO_4$ was kept at 800 °C for 10 hours and melted. After the examining of the X-ray powder diagram of the resulting powder, it was observed that the powder pattern belongs to $SrTeO_3$ rather than $SrTeO_4$. As a result of these studies, it is understood that, $SrTeO_4$ decomposes to $SrTeO_3$ at 720 °C.

Table 2. Crystal data and powder diffraction pattern for Sr TeO_4 Space Group: $Pca2_1$ or Pcam, Z = 4, Mw = 279.22 a.m.u.

a = 5.571(1) b = 13.114 (2) c = 5.002 (1)

V = 365.4 Å^3 D_x = 5.07 gr/cm^2 Dm = 5.08 gr/cm^3

h	k	l	d_{exp}	dcalc	I/I_0	(2θ)exp	(Δ2θ)
0	2	0	6.565	6.557	14	6.206	−0.0073
1	1	0	5.143	5.127	47	7.924	−0.0243
0	2	1	3.977	3.977	6	10.252	0.0000
1	1	1	3.580	3.581	100	11.394	0.0029
1	3	0	3.442	3.439	29	11.852	−0.0101
0	4	0	3.279	3.278	6	12.442	−0.0029
1	3	1	2.837	2.834	83	14.392	−0.0148
2	0	0	2.787	2.785	39	14.650	0.0094
0	4	1	2.743	2.742	43	14.886	−0.0059
0	0	2	2.502	2.501	45	16.330	−0.0048
1	4	1	2.460	2.460	4	16.610	0.0003
1	5	0	2.373	2.373	11	17.224	−0.0011
2	2	1	2.282	2.282	3	17.916	−0.0050
1	1	2	2.248	2.248	7	18.190	0.0003
1	3	2	2.023	2.023	18	20.232	−0.0033
0	6	1	2.002	2.003	3	20.448	0.0086
0	4	2	1.989	1.989	5	20.584	−0.0032
2	4	1	1.954	1.954	50	20.954	−0.0013
2	0	2	1.861	1.861	22	22.016	0.0004
2	2	2	1.791	1.790	3	22.886	−0.0107
3	1	1	1.726	1.726	8	23.762	−0.0044
1	5	2	1.722	1.722	11	23.818	−0.0070
2	3	2	1.710	1.712	5	23.988	0.0331
1	7	1	1.674	1.673	4	24.512	−0.0085
0	8	0	1.639	1.639	6	25.044	0.0043
2	6	1	1.626	1.626	3	25.246	0.0006
2	4	2	1.618	1.618	1	25.374	0.0075
1	1	3	1.586	1.586	10	25.894	0.0045

REFERENCES

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