

In 1912 the German physicist von Laue took up the problems and reasoned that, if crystals were composed of regularly spaced atoms which might act as scattering centre for X-rays and X-rays were electromagnetic waves of wavelength about equal to the interatomic distance in crystals, then it should be possible to diffract X-rays by means of crystals. Under his direction, Friedrich and Kuipping conducted experiments to test this hypothesis. A crystal of copper sulphate was set in the path of a narrow beam of X-rays and a photographic plate was arranged to record the presence of diffracted beam if any. The second attempt was successful and showed without doubt that X-rays were diffracted by crystal out of the primary beam to form a pattern of spots on the photographic plate. For many years, mineralogists and crystallographers have accumulated knowledge about crystals, chiefly by measurement of interfacial angles, chemical analysis and determination of physical properties.

The two English physicists, Bragg (1862-1942) and his son Bragg (1890-1971) were analyzed the above experiments carefully and latter although only a young student at that time it was still the year 1912 successfully was able to express the necessary conditions for diffraction and in a considerably simple mathematical form and is also called Bragg's Law¹.

$$n\lambda = 2d \sin\theta \quad \dots (1)$$

Where λ is the wave length whose coefficient is now unity and n is the order of diffraction the smallest value of n is 1. $n=0$ corresponds to the beam diffracted in the same direction as the transmitted beam, it cannot be observed. The θ is an angle between the diffracted beam and the transmitted beam and is also known as the diffraction angle, 'd' interplanar distance between the two atoms.

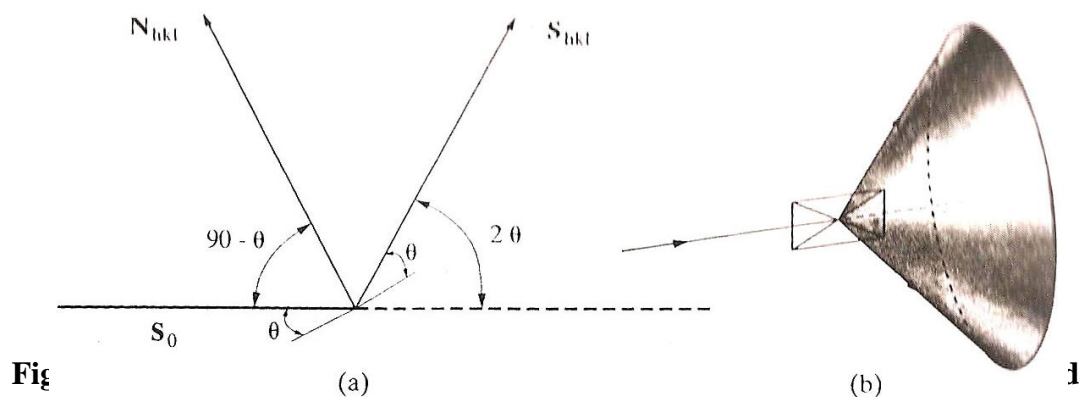
Diffraction can occur wherever Bragg's law. $n\lambda = 2d \sin\theta$ is satisfied. This equation puts very stringent conditions on λ and θ for any given crystal. To satisfy Bragg's law must be devised and this can be done by continuously varying either λ or θ during the experiment. The ways in which these quantities are varied distinguish three main diffraction methods.

1. Laue method
2. Rotating single crystal method
3. Powder method

Powder Method

In the powder method the crystal to be examined is reduced to a very fine powder or already in the form of loose or consolidated microscopic grains. The sample in a suitable holder is placed in a beam of monochromatic X-rays. Each particle of the powder is a tiny crystal, or assemblage of smaller crystals, oriented at random with respect to the incident beam. Just by chance, some of the crystals will be correctly oriented so that their (1 0 0) planes for example can diffract the incident beam. Some other crystals will be correctly oriented for (1 1 0) reflection and so on. The result is that every set of lattice planes will be capable of diffraction^{2,3}. The powder is equivalent in fact, to a single crystal rotated, not about one axis, but also about all possible axis.

Consider one particular hkl reflection and remember that S , S_0 and N_{hkl} , the normal to the diffraction planes (hkl), must be coplanar. One or more little crystals will, by chance, be so oriented that their (hkl) planar makes the correct Bragg's angle for diffraction. Fig. 4 (a) shows one plane in this set and the diffracted beam formed. If this plane is now rotated about the incident beam in such a way that θ is kept constant, then the diffracted beam will travel over the surface of cone as shown in fig. 4 (b).



The axis of the cone coinciding with the transmitted beam. Equivalently, one can imagine rotating N_{hkl} about S_0 while keeping the angle between them is equal to 90° .

Conversely, if the shape and size of the unit cell of the crystal are known, the position of all possible diffraction lines on the film can be predicted. The line of lowest 2θ value is produced by diffraction from planes of the greatest spacing. In the

cubic system, for example d is maximum when $(h^2+k^2+l^2)$ is minimum, and the minimum value of this term is 1, corresponding to $(h\ k\ l)$ equal to $(1\ 0\ 0)$. The $(1\ 0\ 0)$ reflection is accordingly the one of lowest 2θ value. The next possible reflection will have indices $(h\ k\ l)$ corresponding to the next higher value of $(h^2+k^2+l^2)$, namely 2, in which case $(h\ k\ l)$ equals $(1\ 1\ 0)$ and so on.

For a cubic crystal, values of $\sin^2\theta$ are given by equation 4(2).

$$\sin^2\theta = \frac{\lambda^2}{4a^2} (h^2 + k^2 + l^2) \quad \dots(2)$$

By known values of $\sin^2\theta$, λ and $(h^2+k^2+l^2)$ the lattice constant

$$a = \sqrt{\frac{\lambda^2}{4\sin^2\theta} (h^2 + k^2 + l^2)} \quad \dots(3)$$

Where $\lambda = 1.54056\ \text{\AA}$ is a constant for Cu-K_α X-rays

Due to practical difficulties in obtaining good crystals, single crystal XRD could not be employed ultimately to confirm the structure. However, powder X-ray diffractogram is some consolation to us.

For the present study of powder X-ray diffraction data were recorded on a diffractometer attached to a Phillips X-ray machine using $\text{Cu-K}_\alpha(1.54056\text{\AA})$ radiation at SAIF/STIC, Cochin.

4. Results and discussion of X-ray powder diffraction studies

Due to practical difficulties in obtaining good crystals, single crystal XRD could not be employed ultimately to confirm the structure of all the compounds. Powder X-ray diffraction studies of the ligand [BCAP] and its copper complex have been recorded with Cu-K_α X-ray source in the range $3-80^\circ$. All the remaining compounds were found to be amorphous. It is not possible, to get single crystal of any of the complexes and all our efforts to set single crystal were in vain.

4.1. X-ray powder diffraction of ligand [BCAP]

Powder X-ray diffraction pattern of the ligand [BCAP] has been depicted in Fig 4(1). From the figure it is clear that the ligand [BCAP] consists of 10 reflections in the range $11-28^\circ$ (2θ) arising from the diffraction of X-rays by the planes of the compound. The inter-planar spacing (d) has been calculated from the positions of intense peaks using Bragg's relation,

$$n\lambda = 2d\sin\theta,$$

Where λ - wavelength of X-ray used (Cu K_{α} = 1.5406 Å).

The calculated spacing together with relative intensities with respect to most intense peaks are recorded in the Table 4(1). The 2θ value with maximum intensity of the peak for this ligand was found to be 23.674° (2θ), which correspond to $d=3.755$ Å. 2θ values for all the prominent peaks have been listed in the Table 4(1). All the important peaks have been indexed and the observed values of inter-planar distances have been compared with the calculated ones. It was found that there is a good agreement between the calculated and observed values. The unit cell calculations have been carried out for the cubic symmetry of the compound. The characteristic of cubic system is that $\sin^2\theta$ values have a common factor. The cell parameter has been calculated by using the equation,

$$\sin^2\theta = \lambda^2/4a^2 (h^2+k^2+l^2), \text{ where, } \lambda^2/4a^2 \text{ is the common factor.}$$

In the present case $\lambda^2/4a^2$ is common factor.

The experimental values of $\sin^2\theta/\text{common factor}$ are recorded for each peak in the Table 4(1). The $(h^2+k^2+l^2)$ values are 1, 2, 3, 4, 5 and 6. The absence of forbidden numbers confirms the non cubic symmetry.

a. X-ray powder diffraction studies of the Cu(II) complex of the ligand [BCAP]

XRD patterns for Cu(II) complex of ligand [BCAP] have been depicted in Fig.4(2). The diffraction pattern for the Cu(II) complex showed 9 reflections in the range $6-27^{\circ}$ (2θ) arising from the diffraction of X-rays by planes of complex. The inter planar spacing 'd' has been calculated from the positions of intense peaks using the Bragg's relation $n\lambda=2d\sin\theta$. The calculated inter planar spacing together with relative intensities with respect to most intense peaks are recorded in Table 4(2). The 2θ values with maximum intensities of the peak for the Cu(II) complexes was found to be 7.983° (2θ) which correspond to $d=11.066$ Å. All the important peaks have been indexed and the observed values of inter planar distances have been compared with the calculated ones. It was observed that there is a good agreement between the calculated and observed values⁴.

The experimental values of $\sin^2\theta/\text{common factor}$ for Cu(II) complex is recorded for each peak in the Table 4(2). The $(h^2+k^2+l^2)$ values obtained for Cu(II) complex were 1,2,3,4,5,6, 14, and 15. The presence of forbidden numbers 15, indicate that Cu(II) complex may belong to cubic system. The experimental values are in good agreement with $(h^2+k^2+l^2)$ values of primitive type cubic cell with lattice parameter equal to, $a=b=c=14.321 \text{ \AA}$.

4.2. Powder X-ray diffraction data of the ligand [BCAT]

X-ray diffraction pattern for the ligand [BCAT] and Ni(II) complex have been recorded with Cu- K_{α} X-ray source in the range $3-80^{\circ}$. Powder X-ray diffraction pattern of the ligand [BCAT] have been depicted in Fig.4(3), from the figure it is clear the ligand [BCAT] shows eleven reflections in the range $8-35^{\circ}$ (2θ) arising from the diffraction of X-rays by planes of compounds. The interplanar spacing 'd', has been calculated from the position of intense peaks using Bragg's relation. The calculated spacing together with relative intensities with respect to most intense peaks has been recorded in Table 4(3).

The 2θ values with maximum intensity of the peak for the compound was found to be 26.652° (2θ) which corresponds to $d = 3.341 \text{ \AA}$. The 2θ values for the prominent peaks have been listed in the Table 4(3). The experimental values of $\text{Sin}^2\theta/\text{common factor}$ are recorded for each peak. The $(h^2 + k^2 + l^2)$ value are 1, 2, 3, 4, 5, 6, 8, 9, 10, 13 and 16. The absence of forbidden number indicates the ligand [BCAT] may belongs to non cubic symmetry.

a. Powder X-ray diffraction data of Ni(II) complex of the ligand [BCAT]

Powder XRD patterns for Ni(II) complex of ligand [BCAT] have been depicted in Fig 4(4). The Diffraction pattern for the Ni(II) complex showed 6 reflections in the range 6-50° (2θ) arising from the diffraction of X-rays by planes of Ni(II) complex, The inter-planar spacing 'd' has been calculated from the positions of intense peaks using the Bragg's relation $n\lambda=2d\sin\theta$. The calculated inter planar spacing together with relative intensities with respect to most intense peaks are recorded in Table 4(4). The 2θ values with maximum intensities of the peak for the complex was found to be 31.907° (2θ), that correspond to d=2.802 Å. All the important peaks have been indexed and the observed values of inter planar distances have been compared with the calculated ones. It was observed that there is good agreement between the calculated and observed values⁵.

The experimental values of $\sin^2\theta$ /common factor are recorded for each peak in the Tables 4(4). The $(h^2+k^2+l^2)$ values obtained were 1, 2, 3, 4, 5 and 9. The absence of forbidden numbers indicated the compounds may belong to non cubic system.

4.3 Powder X-ray diffraction data of the ligand [BCACP]

Powder XRD patterns of ligand [BCACP] and its Zn(II) complex have been recorded with Cu-K_α X-ray source in the range 3-80°. Powder X-ray diffraction pattern of the ligand [BCACP] has been depicted in Fig. 4(5). From the figure it is clear that the ligand consists of 12 reflections in the range 11-31°(2θ) arising from the diffraction of X-rays by the planes of the compound. The inter planar spacing (d) has been calculated from the positions of intense peaks using Bragg's relation,

$$n\lambda=2d\sin\theta, \quad \text{where, } \lambda\text{- wavelength of X-ray used (Cu K}_{\alpha}\text{=1.5406 \AA}^{\circ}\text{)} .$$

The calculated spacing together with relative intensities with respect to most intense peaks are recorded in the Table 4(5). The 2θ value with maximum intensity of the peak for this ligand is found to be 22.308° (2θ), which correspond to d=3.982 Å. The 2θ values for the prominent peaks have been listed in the Table 4(5). All the important peaks have been indexed and the observed values of inter planar distances have been compared with the calculated ones. It was found that there is a good agreement between the calculated and observed values. The unit cell calculations have been carried out for the cubic symmetry of the compound. The characteristic of cubic system is that $\sin^2\theta$ values have a common factor. The cell parameter has been

calculated by using the equation for cubic system, $\sin^2\theta = \lambda^2/4a^2 (h^2+k^2+l^2)$, where, $\lambda^2/4a^2$ is the common factor.

The experimental values of $\sin^2\theta$ /common factor are recorded for each peak in the table. The $(h^2+k^2+l^2)$ values are 1, 2, 3, 4, 5, 6 and 8. The absences of forbidden numbers indicate that the compound may belong to non cubic system.

a. Powder X-ray diffraction data of Zn(II) complex of the ligand [BCACP]

The XRD patterns for Zn(II) complex of ligand [BCACP] have been depicted in Fig.4(6). The diffraction pattern for the Zn(II) complex showed 13 reflections in the range 7-54° (2θ) arising from the diffraction of X-rays by planes of complex. The inter planar spacing 'd' has been calculated from the positions of intense peaks using the Bragg's relation $n\lambda=2d\sin\theta$. The calculated inter planar spacing together with relative intensities with respect to most intense peaks in case of complex is recorded in Table.4(6). The 2θ values with maximum intensity of the peak for the Zn(II) complex was found to be 26.942° correspond to $d=3.066 \text{ \AA}$. All the important peaks have been indexed and the observed values of inter planar distances have been compared with the calculated ones. It was observed that there is a good agreement between the calculated and observed values.

The experimental values of $\sin^2\theta$ /common factor for both Zn(II) complex was recorded for each peak in the Table 4(6). The $(h^2+k^2+l^2)$ values obtained for Zn(II) complex are 1, 2,3, 4, 5, 6, 8, 9, 10, 13, 14, 18, 22, and 52. The absence of forbidden numbers indicate that Zn(II) complex may belong to non cubic symmetry. The experimental values are in good agreement with $(h^2+k^2+l^2)$ values of primitive type cubic cell with lattice parameter.

4.4 Powder X-ray diffraction data of the ligand [BCMeOACP]

Powder XRD patterns for ligand [BCMeOACP] and its Cd(II) complex have been recorded with Cu-K_α X-ray source in the range 3-80°. Powder X-ray diffraction pattern of the ligand [BCMeOACP] has been depicted in Fig. 4(7). From the figure it is clear that the ligand consists of 5 reflections in the range 24-55°(2θ) arising from the diffraction of X-rays by the planes of the compound. The inter planar spacing (d) has been calculated from the positions of intense peaks using Bragg's relation,

$$n\lambda=2d\sin\theta, \quad \text{where, } \lambda\text{- wavelength of X-ray used (Cu K}_{\alpha}\text{=1.5406 \AA}^{\circ}\text{)}.$$

The calculated spacing together with relative intensities with respect to most intense peaks are recorded in the Table 4(7). The 2θ value with maximum intensity of the peak for this ligand is found to be 26.855° (2θ), which correspond to $d=3.317 \text{ \AA}$. The 2θ values for the prominent peaks have been listed in the Table 4(7). All the important peaks have been indexed and the observed values of inter planar distances have been compared with the calculated ones. It was found that there is a good agreement between the calculated and observed values⁶. The unit cell calculations have been carried out for the cubic symmetry of the compound. The characteristic of cubic system is that $\sin^2\theta$ values have a common factor. The cell parameter has been calculated by using the equation for cubic system, $\sin^2\theta = \lambda^2/4a^2 (h^2+k^2+l^2)$, where, $\lambda^2/4a^2$ is the common factor.

The experimental values of $\sin^2\theta/\text{common factor}$ are recorded for each peak in the table. The $(h^2+k^2+l^2)$ values are 1, 2, 3, 4, and 5. The absences of forbidden numbers indicate that the compound may belong to non cubic system.

a. Powder X-ray diffraction data of Cd(II) complex of the ligand [BCMeOACP]

XRD patterns for Cd(II) complex of ligand [BCMeOACP] have been depicted in Fig.4(8). The diffraction pattern for the Cd(II) complex showed 16 reflections in the range $16-60^\circ$ (2θ) arising from the diffraction of X-rays by planes of complex. The inter planar spacing 'd' has been calculated from the positions of intense peaks using the Bragg's relation $n\lambda=2d\sin\theta$. The calculated inter planar spacing together with relative intensities with respect to most intense peaks in case of both the complex was recorded in Table 4(8). The 2θ values with maximum intensities of the peak for the Cd(II) complexes were found to be 29.804° correspond to $d=2.995 \text{ \AA}$. All the important peaks have been indexed and the observed values of inter planar distances have been compared with the calculated ones. It was observed that there is a good agreement between the calculated and observed values.

The experimental values of $\sin^2\theta/\text{common factor}$ for both Cd(II) complex was recorded for each peak in the Table 4(8). The $(h^2+k^2+l^2)$ values obtained for Cd(II) complex are 1, 2, 3, 5, 7, 8, 9, 10, 11 and 14. The presence of forbidden numbers 7, indicate that Cd(II) complex may belong cubic system. The experimental values are in good agreement with $(h^2+k^2+l^2)$ values of primitive type cubic cell with lattice parameter .

4.5 Powder X-ray diffraction data of the ligand [BCMeTPC]

Powder X-ray diffraction pattern of the ligand [BCMeTPC] and its Ni(II) complex have recorded with Cu-K α X-ray source in the range 3-80 $^{\circ}$. Powder X-ray diffraction pattern of the ligand [BCMeTPC] has been depicted in Fig. 4(9). From the figure it is clear that the ligand it consists of 5 reflections in the range 26-54 $^{\circ}$ (2 θ) arising from the diffraction of X-rays by the planes of the compound. The inter planar spacing (d) has been calculated from the positions of intense peaks using Bragg's relation,

$$n\lambda=2d\sin\theta, \quad \text{where, } \lambda\text{- wavelength of X-ray used (Cu K}\alpha\text{=1.5406 \AA}^{\circ}\text{)}.$$

The calculated spacing together with relative intensities with respect to most intense peaks are recorded in the Table 4(9). The 2 θ value with maximum intensity of the peak for this ligand is found to be 26.908 $^{\circ}$ (2 θ), which correspond to d=3.3108 \AA . The 2 θ values for the prominent peaks have been listed in the Table 4(9). All the important peaks have been indexed and the observed values of inter planar distances have been compared with the calculated ones. It was found that there is a good agreement between the calculated and observed values. The unit cell calculations have been carried out for the cubic symmetry of the compound. The characteristic of cubic system is that sin $^2\theta$ values have a common factor. The cell parameter has been calculated by using the equation for cubic system, sin $^2\theta = \lambda^2/4a^2 (h^2+k^2+l^2)$, where, $\lambda^2/4a^2$ is the common factor.

The experimental values of sin $^2\theta$ /common factor are recorded for each peak in the table. The (h 2 +k 2 +l 2) values are 1, 2, and 4. The absences of forbidden numbers indicate that the compound may belong to non cubic system.

a. Powder X-ray diffraction data of Ni(II) complex of the ligand [BCMeTPC]

XRD patterns for Ni(II) complex of ligand [BCMeTPC] have been depicted in Fig.4(10). The diffraction pattern for the Ni(II) complex showed 4 reflections in the range 20-37 $^{\circ}$ (2 θ) arising from the diffraction of X-rays by planes of complex. The inter planar spacing 'd' has been calculated from the positions of intense peaks using the Bragg's relation $n\lambda=2d\sin\theta$. The calculated inter planar spacing together with relative intensities with respect to most intense peaks in case of both the complex was recorded in Table 4(10). The 2 θ values with maximum intensities of the peak for the Ni(II) complex was found to be 20.1179 $^{\circ}$ correspond to d=4.4118 \AA . All the

important peaks have been indexed and the observed values of inter planar distances have been compared with the calculated ones. It was observed that there is a good agreement between the calculated and observed values.

The experimental values of $\sin^2\theta/\text{common factor}$ for both Ni(II) complex was recorded for each peak in the Table 4(10). The $(h^2+k^2+l^2)$ values obtained for Ni(II) complex are 1, 2, 3 and 4. The absences of forbidden numbers indicate that the compound may belong to non cubic system.

4.6 Powder X-ray diffraction data of the ligand [BCMeTB]

Powder X-ray diffraction pattern of the ligand [BCMeTB] and its Co(II) complex have recorded. Powder X-ray diffraction pattern of the ligand [BCMeTB] have been depicted in **Fig. 4(11)**. From the figure it is clear that the ligand it consists of 5 reflections in the range $10-30^\circ(2\theta)$ arising from the diffraction of X-rays by the planes of the compound. The inter planar spacing (d) has been calculated from the positions of intense peaks using Bragg's relation,

$$n\lambda=2d\sin\theta, \quad \text{where, } \lambda\text{- wavelength of X-ray used (Cu K}_\alpha=1.5406 \text{ \AA}^\circ).$$

The calculated spacing together with relative intensities with respect to most intense peaks are recorded in the Table 4(11). The 2θ value with maximum intensity of the peak for this ligand is found to be $24.398^\circ(2\theta)$, which correspond to $d=3.681 \text{ \AA}^\circ$. The 2θ values for the prominent peaks have been listed in the Table 4(11). All the important peaks have been indexed and the observed values of inter planar distances have been compared with the calculated ones. It was found that there is a good agreement between the calculated and observed values. The unit cell calculations have been carried out for the cubic symmetry of the compound. The characteristic of cubic system is that $\sin^2\theta$ values have a common factor. The cell parameter has been calculated by using the equation for cubic system, $\sin^2\theta = \lambda^2/4a^2 (h^2+k^2+l^2)$, where, $\lambda^2/4a^2$ is the common factor.

The experimental values of $\sin^2\theta/\text{common factor}$ are recorded for each peak in the table. The $(h^2+k^2+l^2)$ values are 1, 4, 6, 7 and 8. The presence of forbidden numbers 7, indicate that Co(II) complex may belong cubic system.

a. Powder X-ray diffraction data of Co(II) complex of the ligand [BCMeTB]

XRD patterns for Co(II) complex of ligand [BCMeTB] have been depicted in **Fig.4(12)**. The diffraction pattern for the Co(II) complex showed 5 reflections in the

range $9-47^\circ$ (2θ) arising from the diffraction of X-rays by planes of complex. The inter planar spacing 'd' has been calculated from the positions of intense peaks using the Bragg's relation $n\lambda=2d\sin\theta$. The calculated inter planar spacing together with relative intensities with respect to most intense peaks in case of both the complex was recorded in Table **4(12)**. The 2θ values with maximum intensities of the peak for the Co(II) complex was found to be 19.986° correspond to $d=4.449 \text{ \AA}$. All the important peaks have been indexed and the observed values of inter planar distances have been compared with the calculated ones. It was observed that there is a good agreement between the calculated and observed values.

The experimental values of $\sin^2\theta/\text{common factor}$ for both Co(II) complex was recorded for each peak in the Table **4(12)**. The $(h^2+k^2+l^2)$ values obtained for Co(II) complex are 1, 4, 7, 8 and 22. The presence of forbidden numbers 7, indicate that Co(II) complex may belong cubic system.

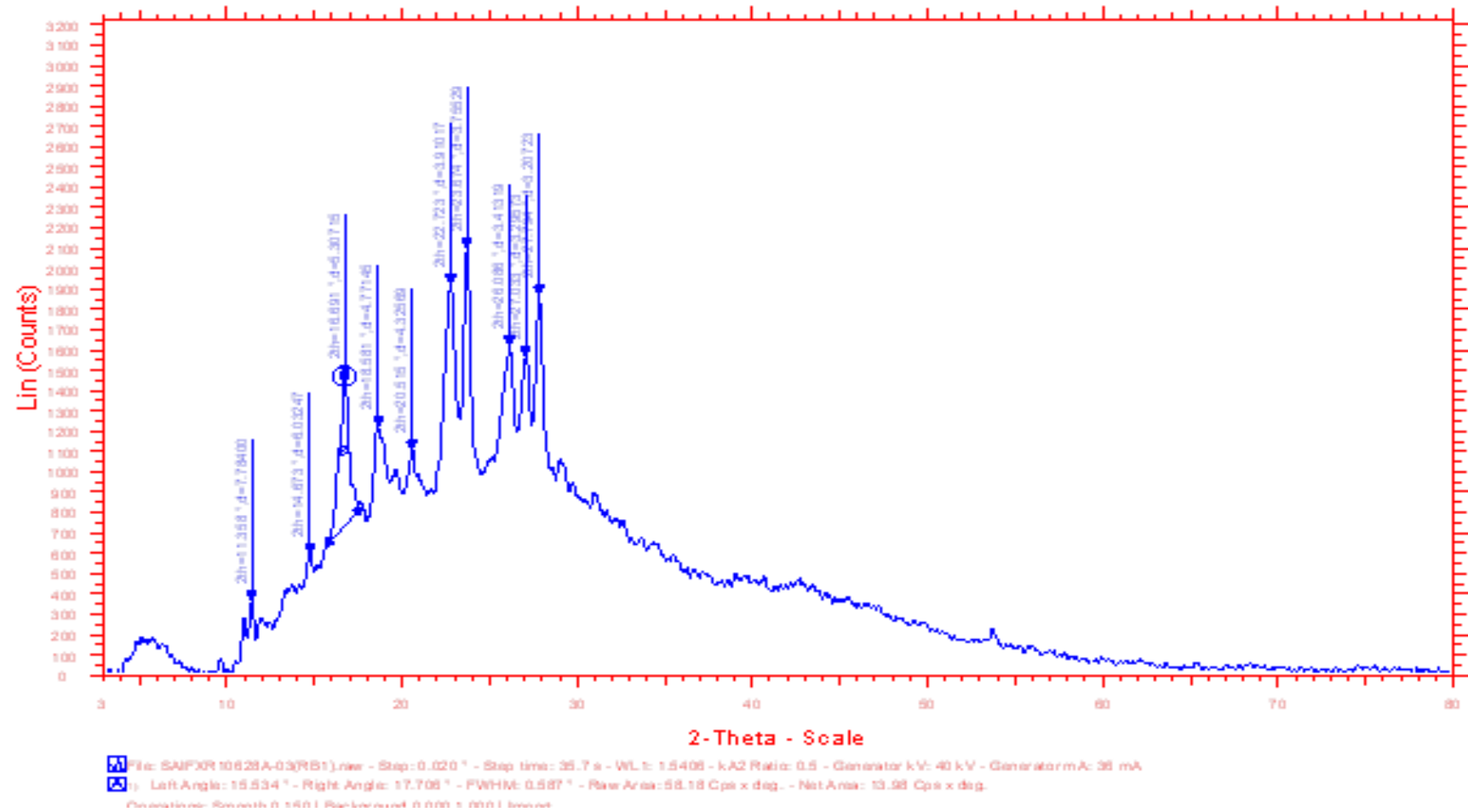


Fig.4 (1) Powder X-ray diffraction pattern of the ligand [BCAP]

Table 4(1)
Powder X-ray diffraction data of the ligand [BCAP]

Sl. No.	2 θ	θ	Sin θ	Sin ² θ	hkl	h ² +k ² +l ²	d		a. in A°
							Calculated	Observed	
1	11.358	5.6790	0.0989	0.0098	1 0 0	1	7.7850	7.7840	7.7840
2	14.673	7.3365	0.1276	0.0163	1 1 0	2	6.0368	6.0347	8.5373
3	15.691	7.8455	0.1365	0.0186	1 1 0	2	5.6432	5.3070	7.9800
4	18.581	9.2905	0.1614	0.0261	1 1 1	3	4.7736	4.7714	8.2664
5	20.515	10.2575	0.1365	0.0186	1 1 0	3	4.3270	4.3250	7.4954
6	22.723	11.3615	0.1969	0.0388	2 0 0	4	3.9121	3.9100	7.8242
7	23.674	11.837	0.2051	0.0420	2 0 0	4	3.7557	3.7552	7.5114
8	26.083	13.0414	0.2256	0.0509	2 1 0	5	3.4144	3.4132	7.6349
9	27.063	13.5315	0.2339	0.0547	2 1 0	5	3.2932	3.2960	7.3640
10	27.794	13.8971	0.2402	0.0576	2 1 1	6	3.2069	3.2072	7.8553

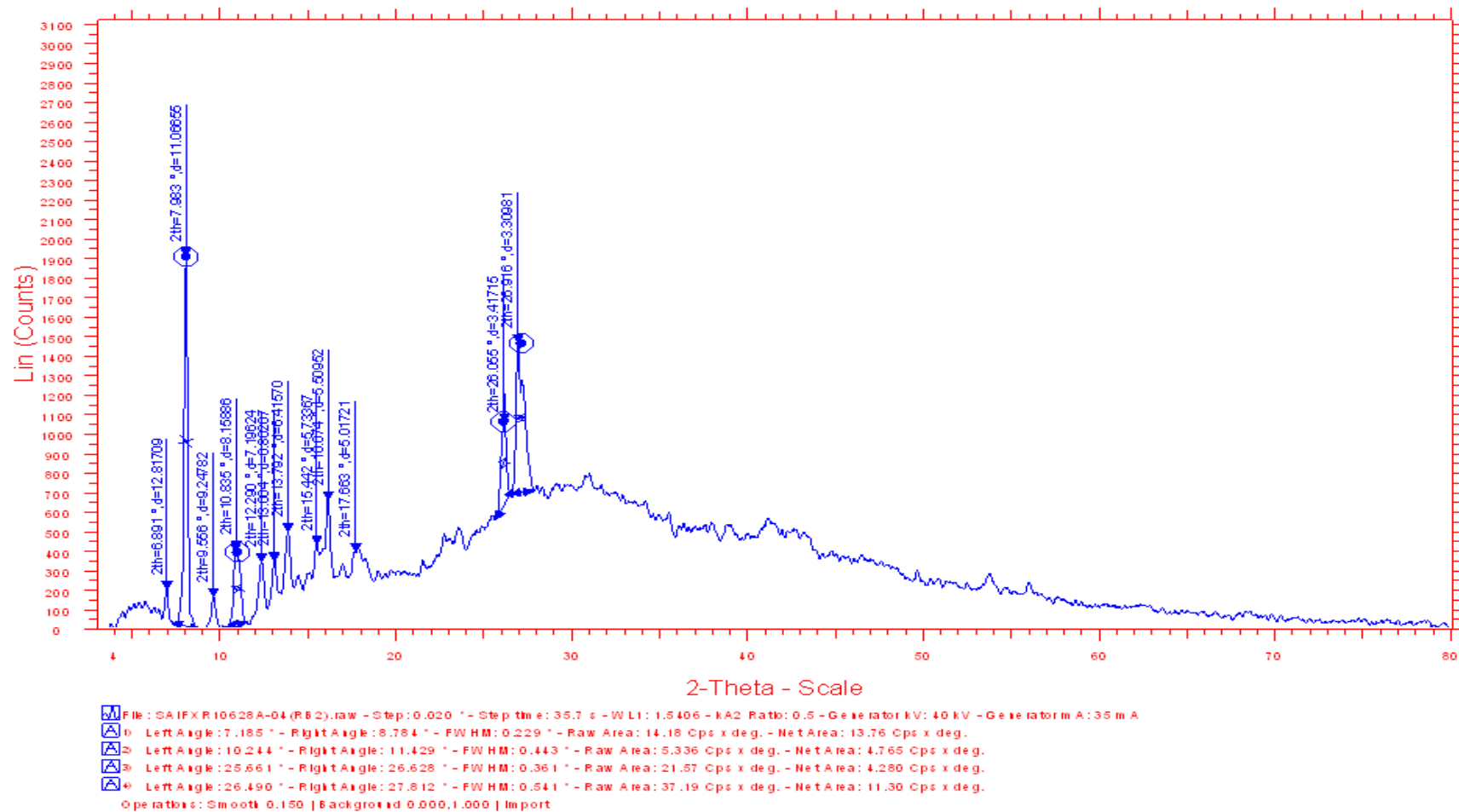
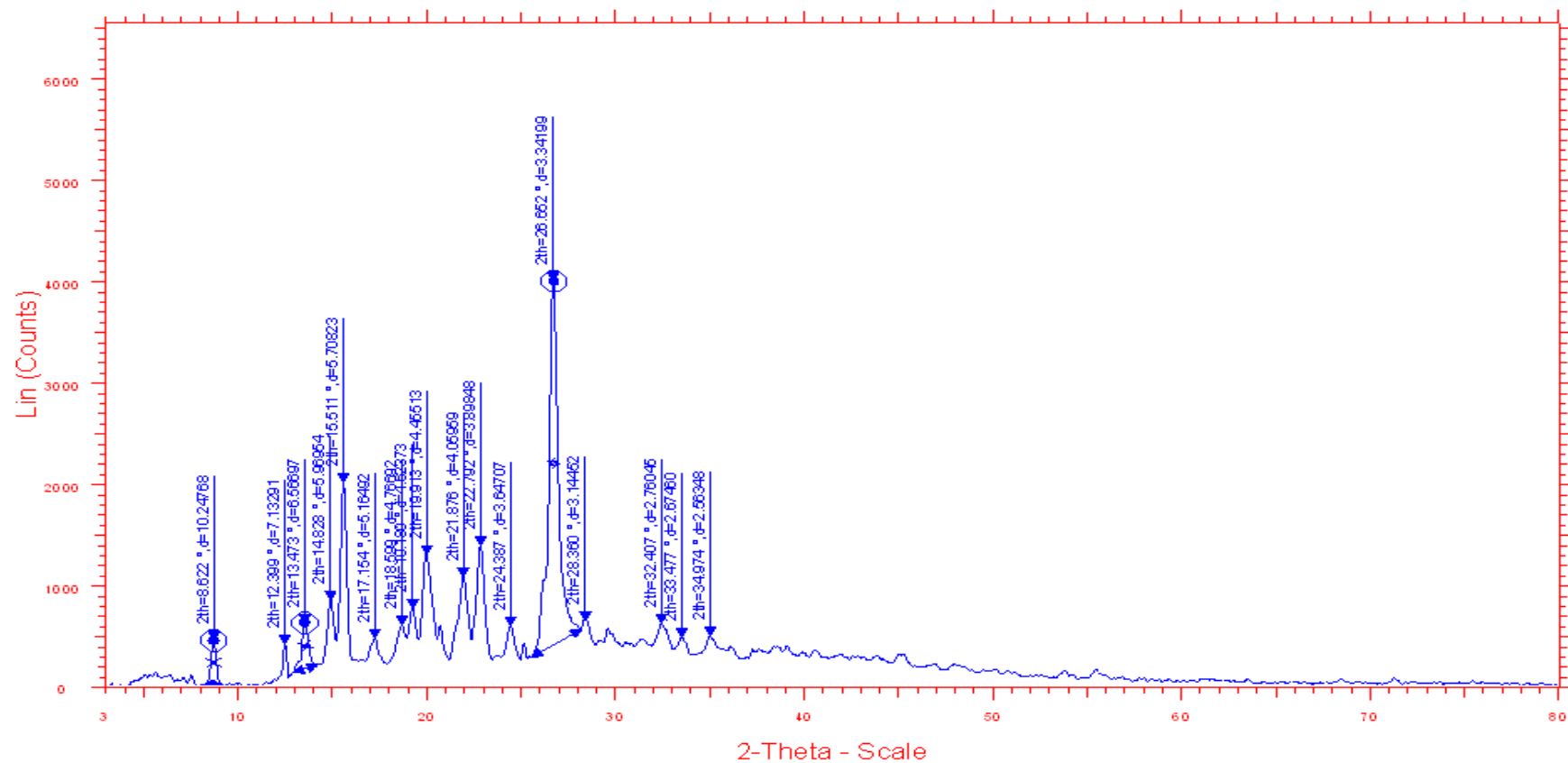


Fig.4 (2) Powder X-ray diffraction pattern of the Cu(II) complex of the ligand [BCAP]

Table 4(2)
Powder X-ray diffraction data of the Cu(II) complex of the ligand [BCAP]

Sl. No.	2θ	θ	Sinθ	Sin ² θ	hkl	h ² +k ² +l ²	d		a. in Å°
							Calculated	Observed	
1	6.891	3.445	0.0600	0.00365	1 0 0	1	12.8383	12.8170	12.8380
2	7.983	3.991	0.0696	0.0048	1 0 0	1	11.0675	11.0665	11.0675
3	9.556	4.7780	0.0833	0.0069	1 1 0	2	9.2474	9.2478	13.0770
4	12.290	6.1450	0.1070	0.0115	1 1 1	3	7.1990	7.1962	12.4690
5	13.792	6.8960	0.1200	0.0142	2 0 0	4	6.4191	6.4157	12.8383
6	15.442	7.7210	0.1343	0.0180	2 1 0	5	5.7356	5.3366	12.8253
7	17.663	8.8315	0.1535	0.0235	2 1 1	6	5.0182	5.0172	13.2770
8	26.055	13.0270	0.2254	0.0508	3 2 1	14	3.4170	3.4171	12.7870
9	26.916	13.4580	0.2327	0.0541	- - -	15	3.3101	3.3098	12.8201



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 Left Angle: 8.073 ° - Right Angle: 9.060 ° - FWHM: 0.308 ° - Raw Area: 4.305 Cps x deg. - Net Area: 3.967 Cps x deg.
 Left Angle: 12.830 ° - Right Angle: 14.153 ° - FWHM: 0.385 ° - Raw Area: 11.86 Cps x deg. - Net Area: 5.417 Cps x deg.
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Fig.4 (3) Powder X-ray diffraction pattern of the ligand [BCAT]

Table 4(3)
Powder X-ray diffraction data of the ligand [BCAT]

Sl. No.	2 θ	θ	Sin θ	Sin ² θ	hkl	h ² +k ² +l ²	d		a. in A°
							Calculated	Observed	
1	8.622	4.3110	0.0752	0.0056	1 0 0	1	10.2434	10.2476	10.2433
2	12.399	6.1999	0.1079	0.01166	1 1 0	2	7.1390	7.1329	10.0945
3	14.828	7.4140	0.1290	0.0166	1 1 1	3	5.9713	5.9695	10.3426
4	17.154	8.5770	0.1491	0.0224	2 0 0	4	5.1663	5.1649	10.3326
5	19.913	9.9565	0.1729	0.0298	2 1 0	5	4.4552	4.4551	9.9630
6	21.873	10.9365	0.1897	0.0359	2 1 1	6	4.0606	4.0595	9.9464
7	24.387	12.1935	0.2112	0.0446	2 2 0	8	3.6472	3.6470	10.3159
8	26.652	13.326	0.2305	0.0531	2 2 1	9	3.3419	3.3419	10.0255
9	28.360	14.1800	0.2449	0.0600	3 1 0	10	3.1453	3.1445	9.9465
10	32.407	16.2035	0.2790	0.0778	3 2 0	13	2.7610	2.7604	9.9548
11	34.974	17.487	0.3004	0.0903	4 0 0	16	2.5642	2.5634	10.2570

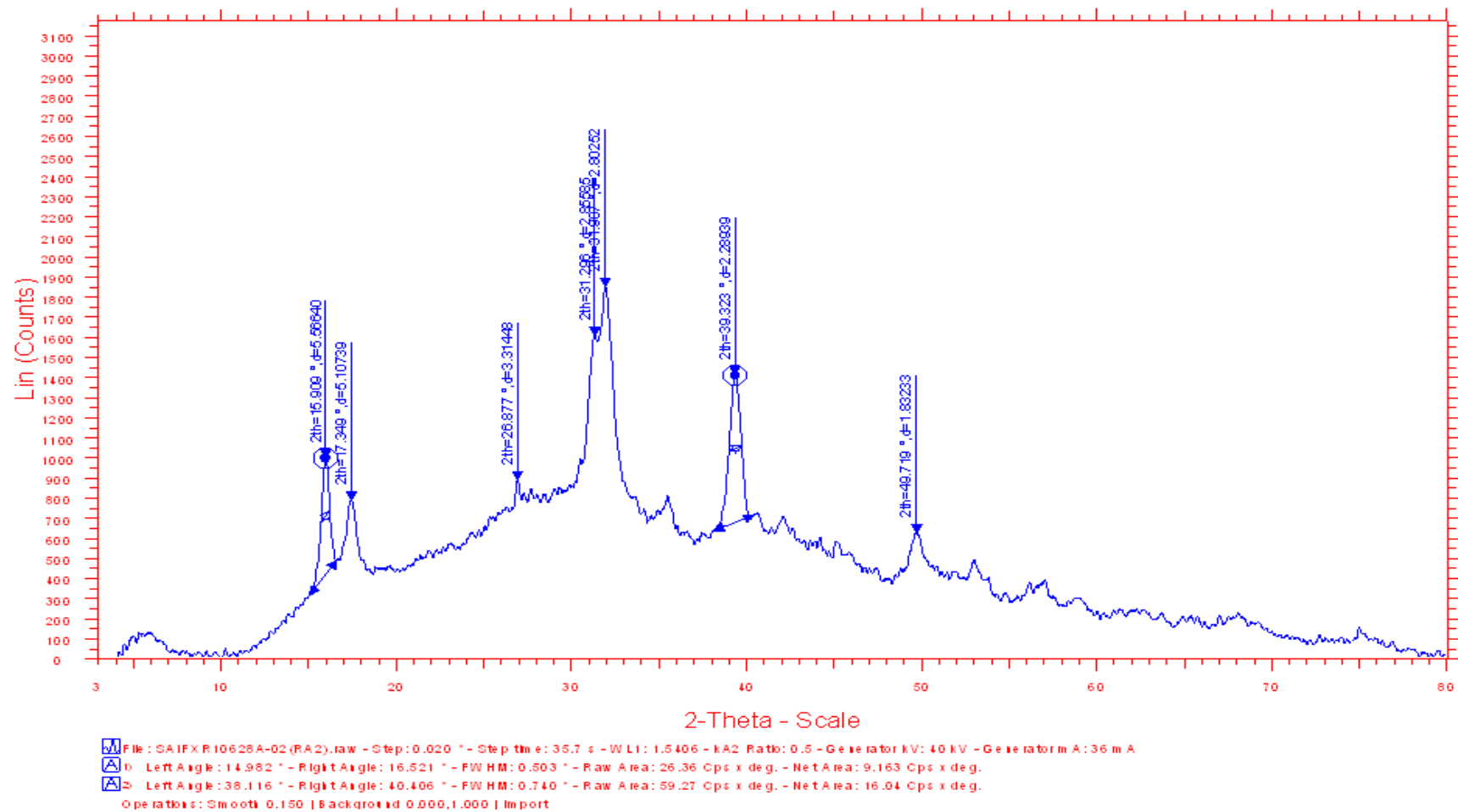


Fig.4 (4) Powder X-ray diffraction pattern of the Ni(II) complex of the ligand [BCAT]

Table 4(4)
Powder X-ray diffraction data of the Ni(II) complex of the ligand [BCAT]

Sl. No.	2θ	θ	Sin θ	Sin ² θ	hkl	$h^2+k^2+l^2$	d		a. in A°
							Calculated	Observed	
1	15.909	7.9545	0.1384	0.0192	1 0 0	1	5.5650	5.5660	5.5657
2	17.349	8.6745	0.1508	0.0227	1 0 0	1	5.1081	5.1074	5.1080
3	26.877	13.4385	0.2324	0.0540	1 1 1	3	3.3140	3.3145	5.7409
4	31.907	15.9535	0.2748	0.0755	2 0 0	4	2.8031	2.8025	5.6062
5	39.323	19.6615	0.3364	0.1132	2 1 0	5	2.2898	2.2894	5.1570
6	49.719	24.859	0.4203	0.1767	2 2 1	9	1.8327	1.8323	5.4982

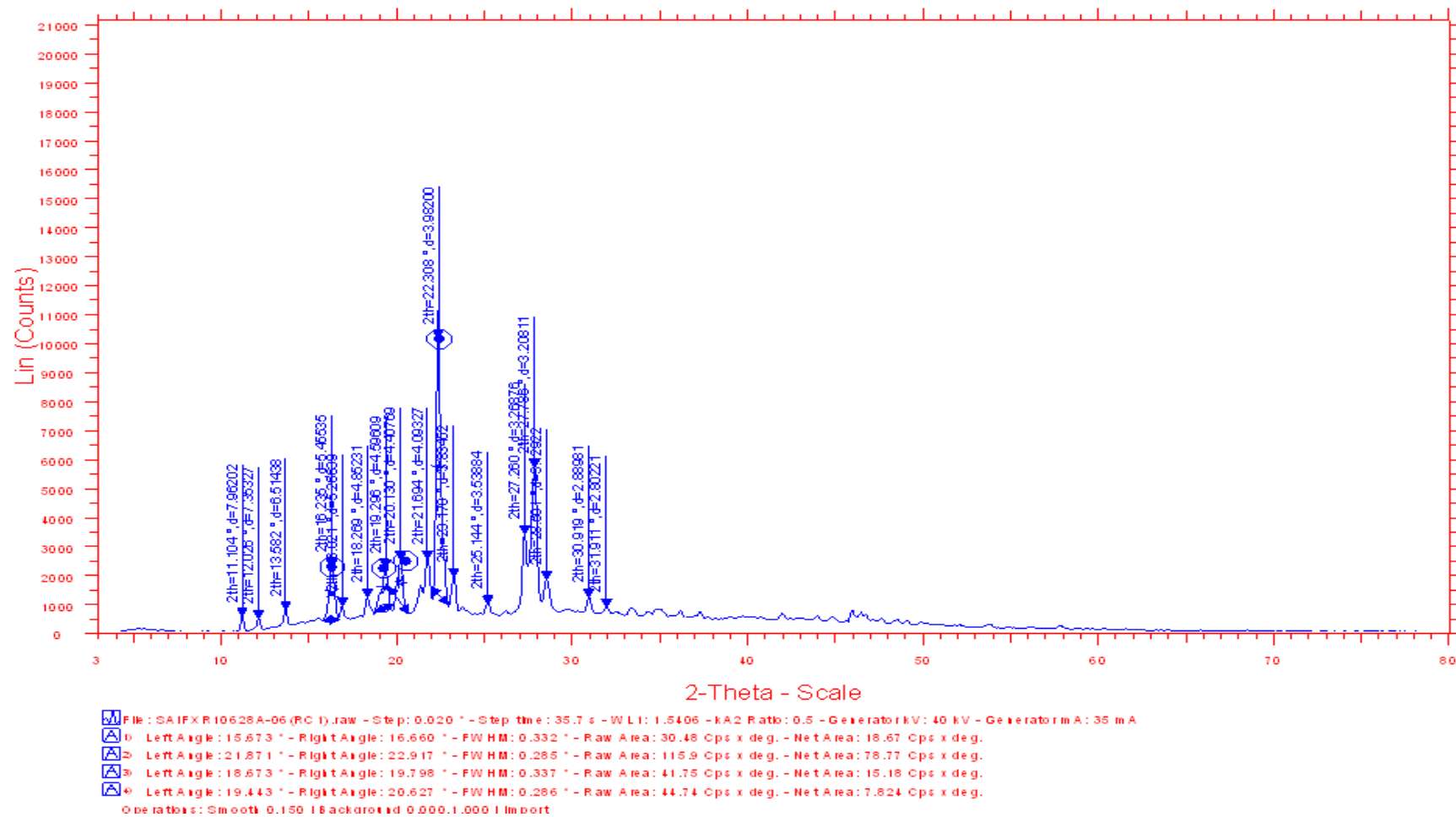


Fig.4 (5) Powder X-ray diffraction pattern of the ligand [BCACP]

Table 4(5)
Powder X-ray diffraction data of the ligand [BCACP]

Sl. No.	2 θ	θ	Sin θ	Sin ² θ	hkl	h ² +k ² +l ²	d		a. in A°
							Calculated	Observed	
1	11.104	5.5520	0.0967	0.0093	1 0 0	1	7.9650	7.9620	7.9658
2	13.582	6.7910	0.1182	0.0139	1 0 0	1	6.5169	6.5143	7.9815
3	16.235	8.3675	0.1455	0.0211	1 1 0	2	5.2941	5.4530	7.4870
4	18.269	9.7345	0.1587	0.0252	1 1 1	3	4.8538	4.8523	8.4070
5	19.296	9.6480	0.1675	0.0280	1 1 1	3	4.5988	4.5960	7.9653
6	20.130	10.065	0.1747	0.0305	1 1 1	3	4.4092	4.4070	7.6370
7	21.694	10.847	0.1881	0.0354	2 0 0	4	4.0951	4.093	8.1903
8	22.308	11.154	0.1934	0.0374	2 0 0	4	3.9829	3.9820	7.9658
9	25.144	12.5720	0.2176	0.0473	2 1 0	5	3.5399	3.5388	7.9134
10	27.260	13.6330	0.2356	0.0555	2 1 1	6	3.2695	3.2687	8.0086
11	30.919	15.4595	0.2665	0.0710	2 2 0	8	2.8904	2.8898	8.1753
12	31.911	15.955	0.2748	0.0755	2 2 0	8	2.8031	2.802	7.9284

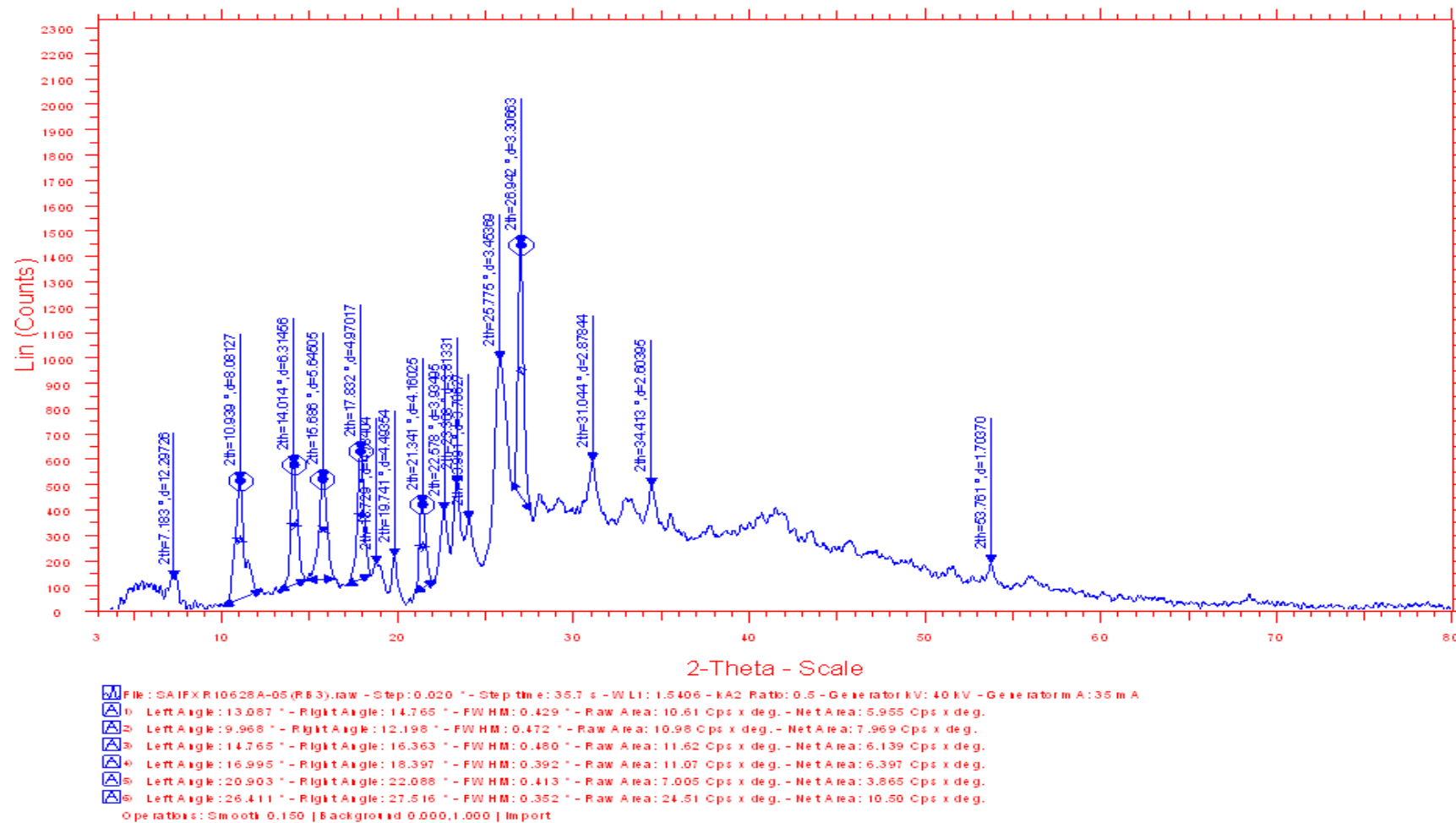


Fig.4 (6) Powder X-ray diffraction pattern of the Zn(II) complex of the ligand [BCACP]

Table 4(6)
Powder X-ray diffraction data of the Zn(II) complex of the ligand [BCACP]

Sl. No.	2θ	θ	Sinθ	Sin ² θ	hkl	h ² +k ² +l ²	d		a. in Å°
							Calculated	Observed	
1	7.183	3.5915	0.0626	0.0039	1 0 0	1	12.2972	12.2973	12.2972
2	10.939	5.4695	0.0953	0.0091	1 1 0	2	8.8020	8.0812	11.4297
3	14.014	7.0070	0.1219	0.0148	2 0 0	4	6.3191	6.3145	12.6382
4	15.686	7.8430	0.1364	0.0186	2 1 0	5	5.6473	5.6451	12.6278
5	7.832	8.9160	0.1549	0.0240	2 1 1	6	4.9728	4.9701	12.1810
6	19.741	9.8705	0.1714	0.0293	2 2 0	8	4.4941	4.4935	12.7114
7	21.341	10.6405	0.1851	0.0343	2 2 1	9	4.1615	4.1602	12.4846
8	23.308	11.6540	0.2020	0.0408	3 1 0	10	3.8133	3.8133	12.0589
9	25.775	12.8875	0.2230	0.0497	3 2 0	13	3.4542	3.4536	12.4545
10	26.942	13.4710	0.2329	0.0542	3 2 1	14	3.3070	3.0663	12.3752
11	31.014	15.5070	0.2673	0.0715	3 3 0	18	2.8818	2.8784	12.2263
12	34.413	17.2065	0.2958	0.0875	3 3 2	22	2.6041	2.6039	12.2144
13	53.761	26.8805	0.4521	0.2044	6 4 0	52	1.7038	1.7037	12.2864

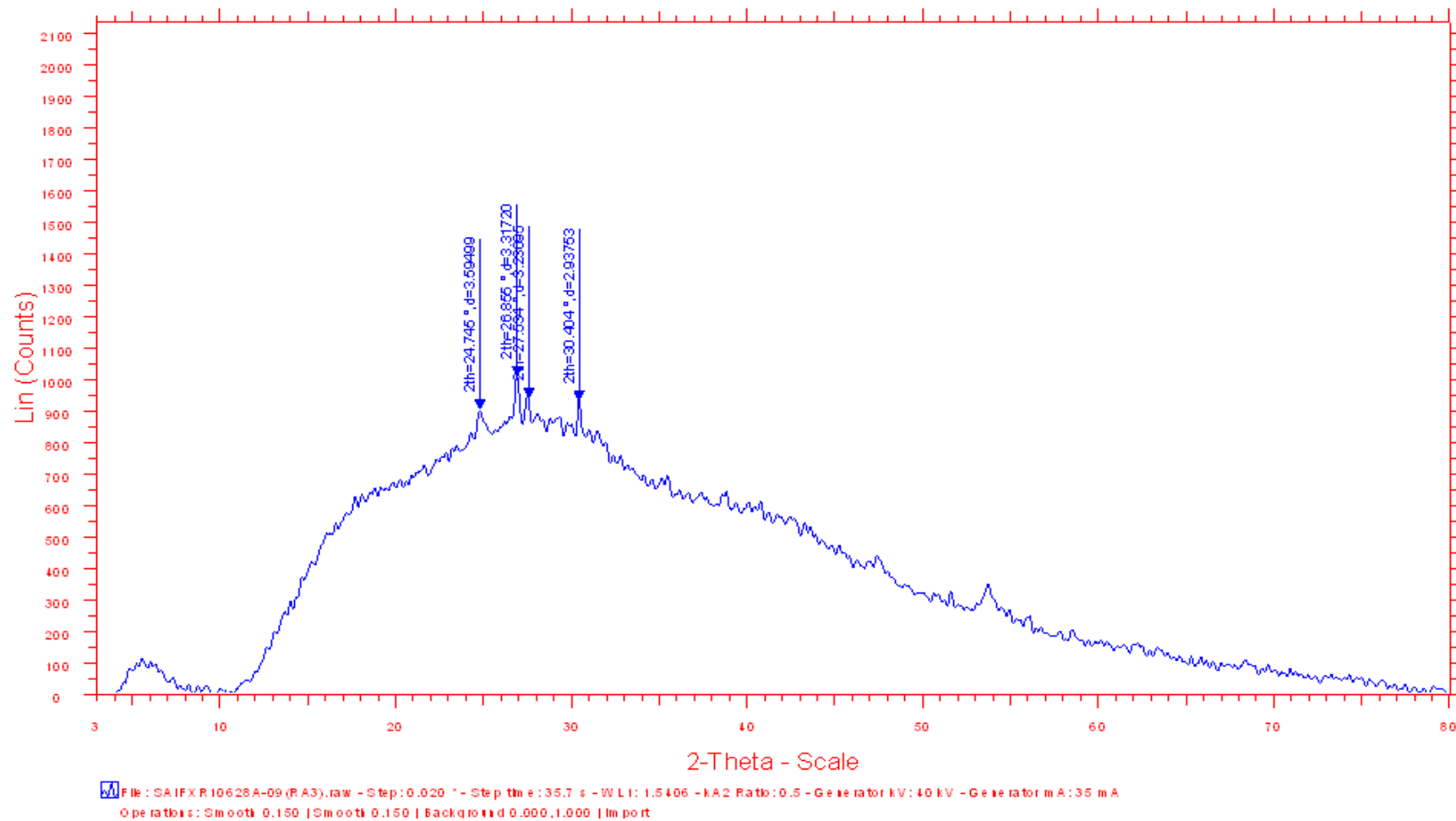


Fig.4 (7) Powder X-ray diffraction pattern of the ligand [BCMeOACP]

Table 4(7)
Powder X-ray diffraction data of the ligand [BCMeOACP]

Sl. No.	2 θ	θ	Sin θ	Sin ² θ	hkl	h ² +k ² +l ²	d		a. in A°
							Calculated	Observed	
1	26.855	13.4275	0.2322	0.0539	1 0 0	1	3.3173	3.3172	3.3173
2	30.404	15.2020	0.2622	0.0687	1 0 0	2	2.9378	2.9375	4.1547
3	40.077	20.0380	0.3426	0.1174	1 1 1	3	2.2483	2.2480	3.8943
4	51.261	25.6301	0.4325	0.1871	2 0 0	4	1.7810	1.7807	3.5620
5	54.583	27.2915	0.4585	0.2102	2 1 0	5	1.6800	1.6797	3.7566

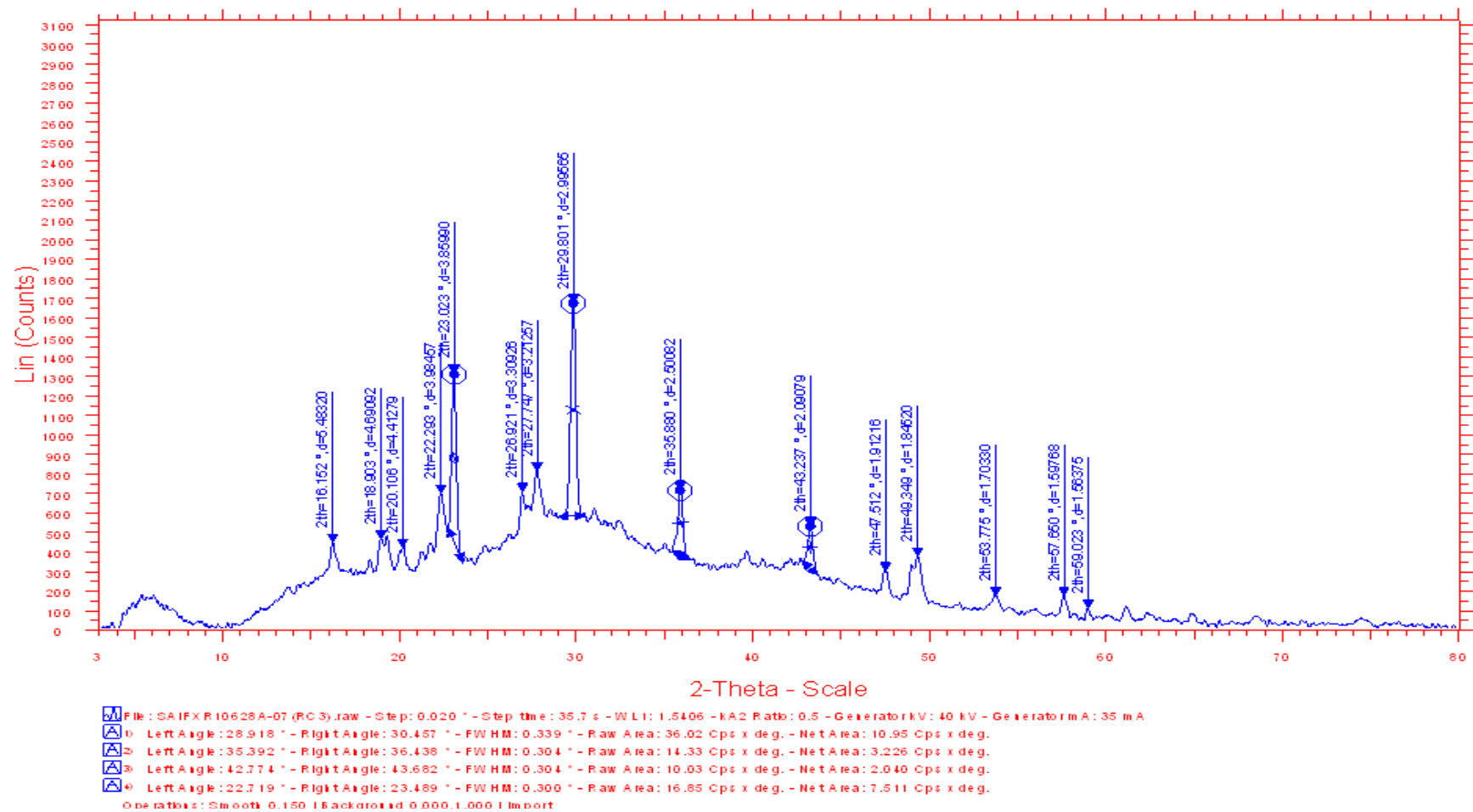


Fig.4 (8) Powder X-ray diffraction pattern of the Cd(II) complex of the ligand [BCMeOACP]

Table 4 (8)
Powder X-ray diffraction data of the Cd(II) complex of the ligand [BCMeOACP]

Sl. No.	2θ	θ	Sinθ	Sin ² θ	hkl	h ² +k ² +l ²	d		a. in Å°
							Calculated	Observed	
1	16.152	8.0760	0.1404	0.0197	1 0 0	1	5.4864	5.4832	5.4804
2	18.903	9.4515	0.1642	0.0269	1 0 0	1	4.6912	4.6920	4.6912
3	20.106	10.0530	0.1744	0.0304	1 1 0	2	4.4128	4.4128	6.2463
4	22.293	11.1465	0.1933	0.0373	1 1 0	2	3.9849	3.9840	5.635
5	23.023	11.5115	0.1995	0.0398	1 1 0	2	3.8611	3.8590	5.4605
6	26.921	13.4605	0.2327	0.0542	1 1 1	3	3.3102	3.3000	5.7335
7	27.747	13.8735	0.2397	0.0574	1 1 1	3	3.2136	3.2120	5.5661
8	29.801	14.900	0.2571	0.0661	1 1 1	3	2.9961	2.9956	5.1894
9	35.880	17.9400	0.3080	0.0948	2 1 0	5	2.5009	2.5008	5.5923
10	43.237	21.6180	0.3684	0.1357	- - -	7	2.0903	2.0908	5.5320
11	47.512	23.7560	0.4028	0.1622	2 2 0	8	1.9123	1.9126	5.4089
12	49.349	24.6745	0.4174	0.1742	2 2 1	9	1.8451	1.8452	5.5364
13	53.775	26.8870	0.0522	0.2045	3 1 0	10	1.7034	1.7033	5.3867
14	57.650	28.8250	0.4821	0.2324	3 1 1	11	1.5978	1.5976	5.2993
15	59.023	29.5115	0.4925	0.2476	3 2 1	14	1.5640	1.5637	5.8521

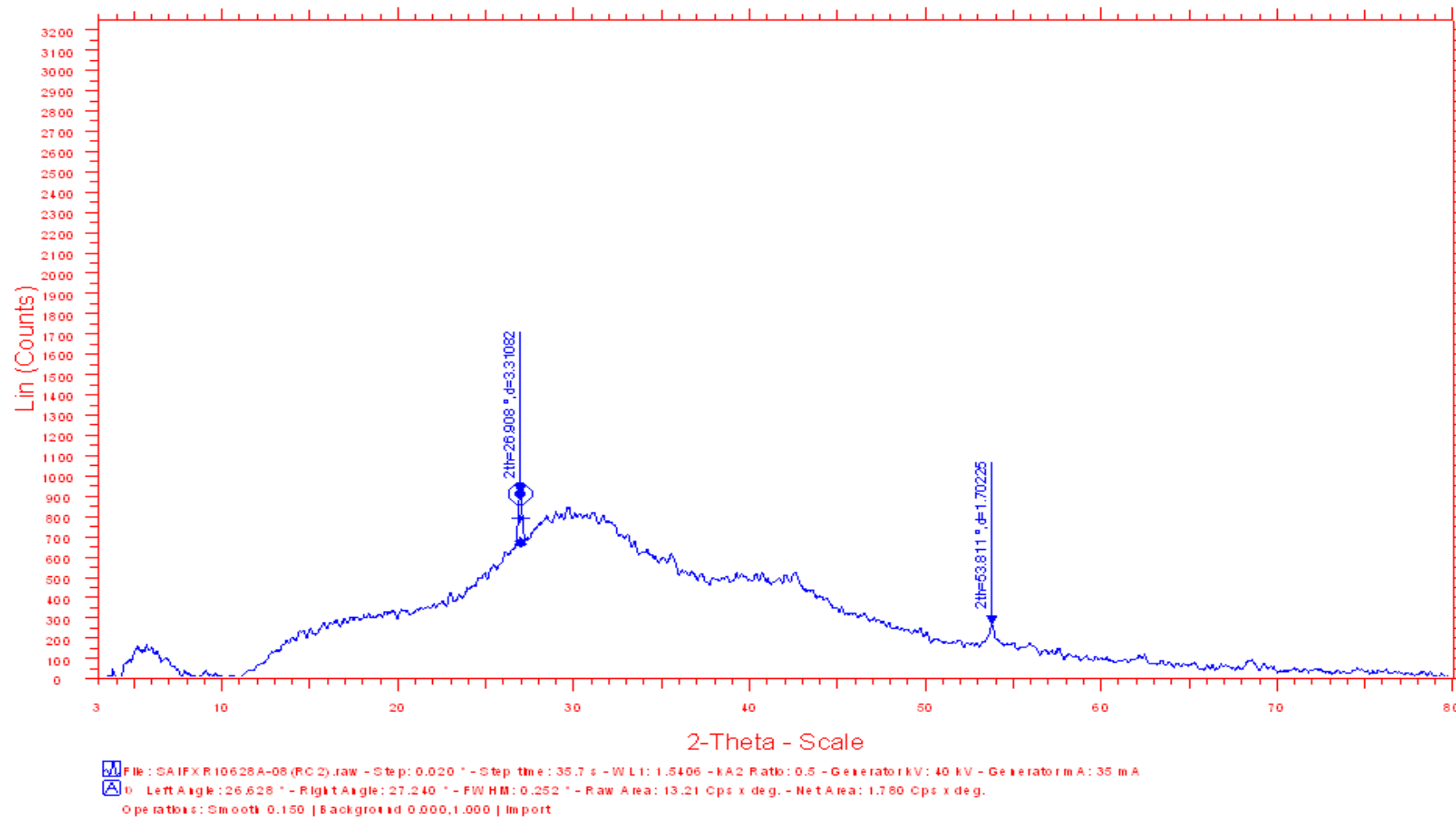


Fig.4 (9) Powder X-ray diffraction pattern of the ligand [BCMeTPC]

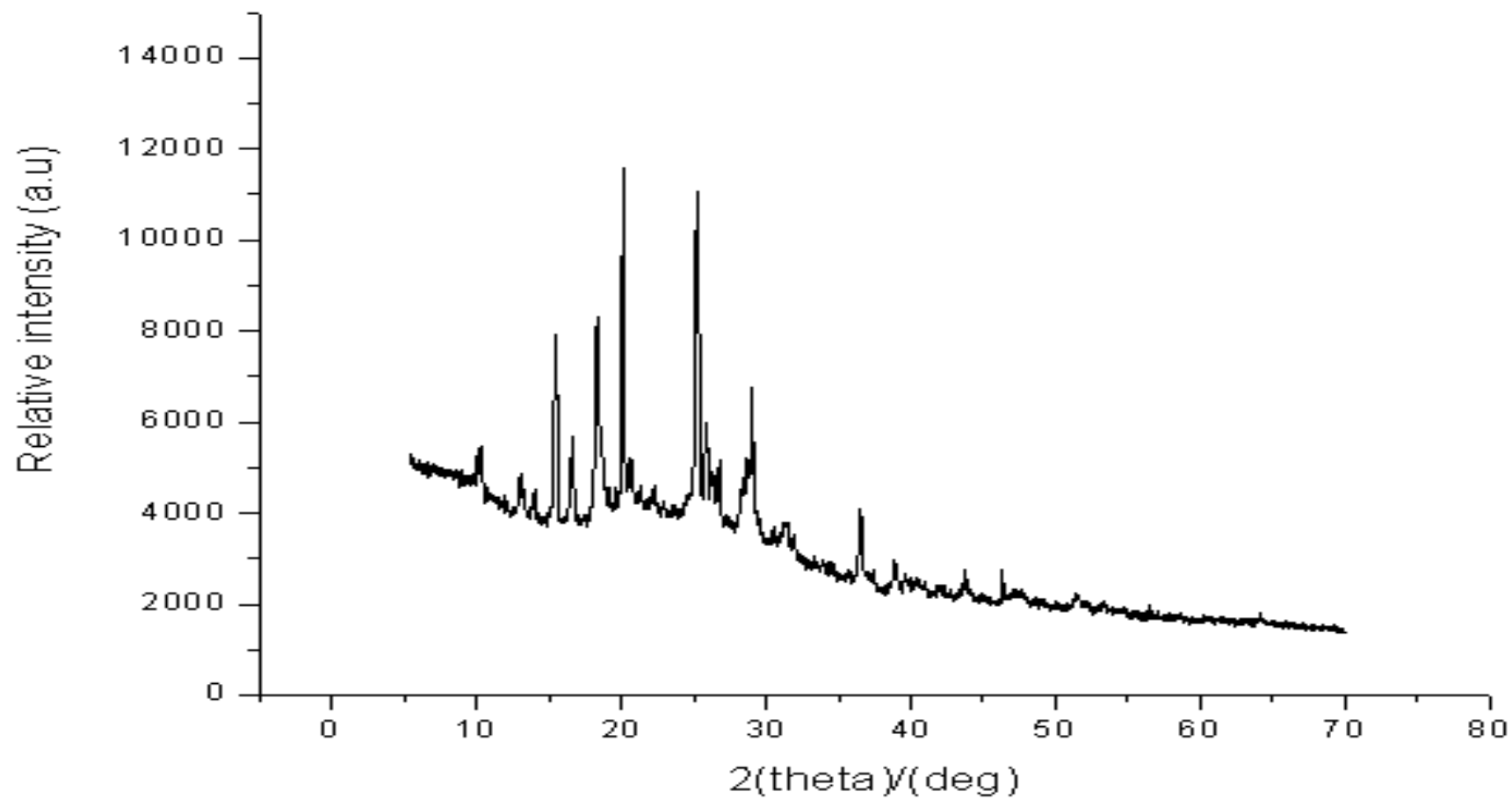


Fig.4 (10) Powder X-ray diffraction pattern of the Ni(II) complex of the ligand [BCMeTPC]

Table 4-(9)
Powder X-ray diffraction data of the ligand [BCMeTPC]

Sl. No.	2θ	θ	Sinθ	Sin ² θ	hkl	h ² +k ² +l ²	d		a. in Å°
							Calculated	Observed	
1	23.540	11.7700	0.2039	0.0416	1 0 0	1	3.7778	3.7856	3.7778
2	26.908	13.4540	0.2326	0.0541	1 0 0	1	3.3116	3.3108	3.1170
3	35.155	17.5770	0.3019	0.0912	1 1 0	2	2.4024	2.5507	3.6083
4	43.486	21.1743	0.3704	0.1373	1 0 0	2	2.0626	2.0796	3.0100
5	53.811	26.9055	0.4525	0.2048	2 0 0	4	1.7023	1.7022	3.4046

Table 4(10)
Powder X-ray diffraction data of the Ni(II) complex of the ligand [BCMeTPC]

Sl. No.	2θ	θ	Sinθ	Sin ² θ	hkl	h ² +k ² +l ²	d		a. in Å°
							Calculated	Observed	
1	20.1179	10.058	0.1746	0.0305	1 0 0	1	4.4120	4.4117	4.4118
2	25.2650	12.6330	0.2187	0.0478	1 1 0	2	3.5222	3.5220	4.9811
3	29.0465	14.5230	0.2508	0.0629	1 1 1	3	3.0738	3.0690	5.3955
4	36.5571	18.2790	0.3136	0.0984	2 0 0	4	2.4563	2.4490	4.4126

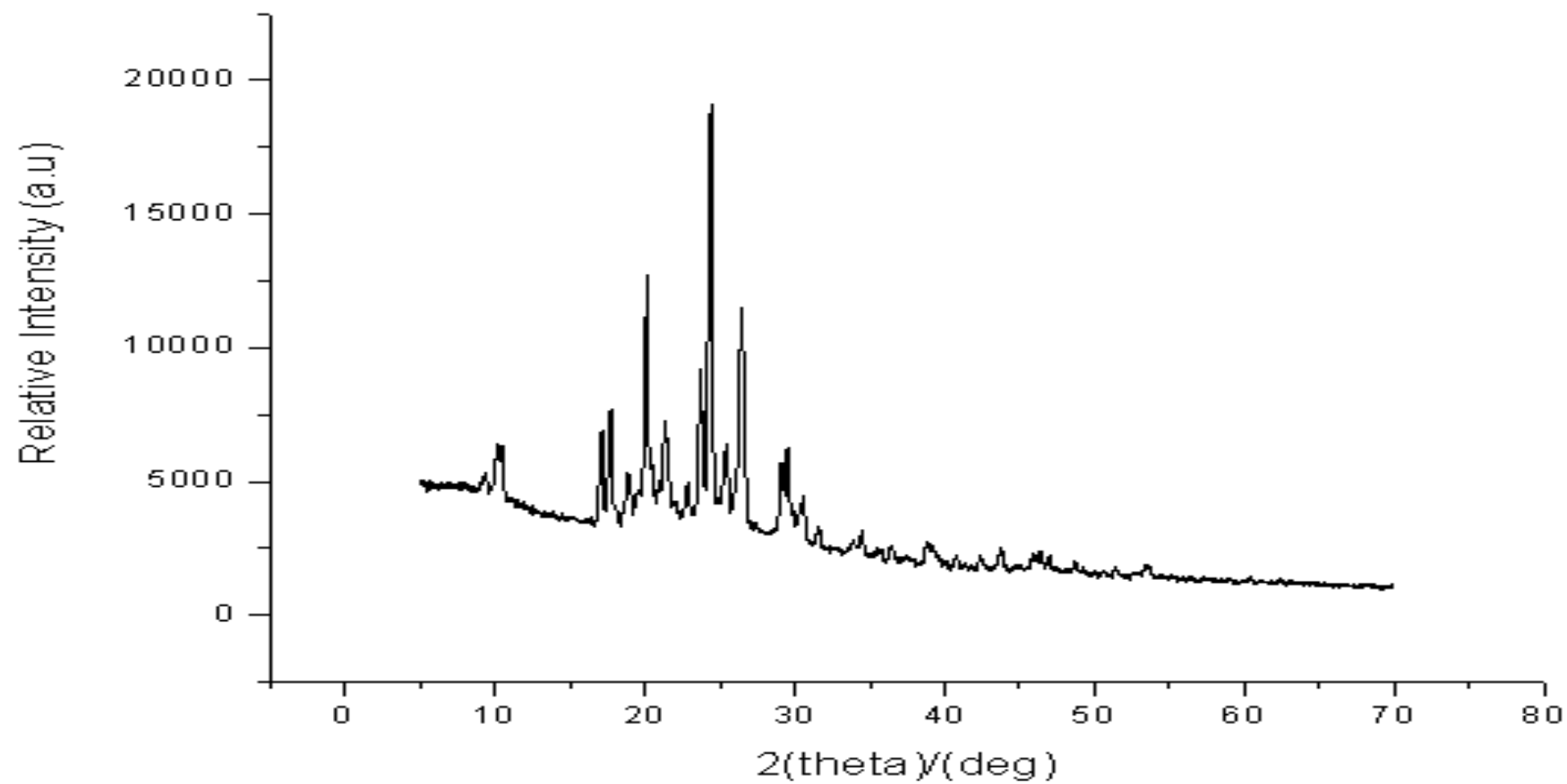


Fig.4 (11) Powder X-ray diffraction pattern of the ligand [BCMeTB]

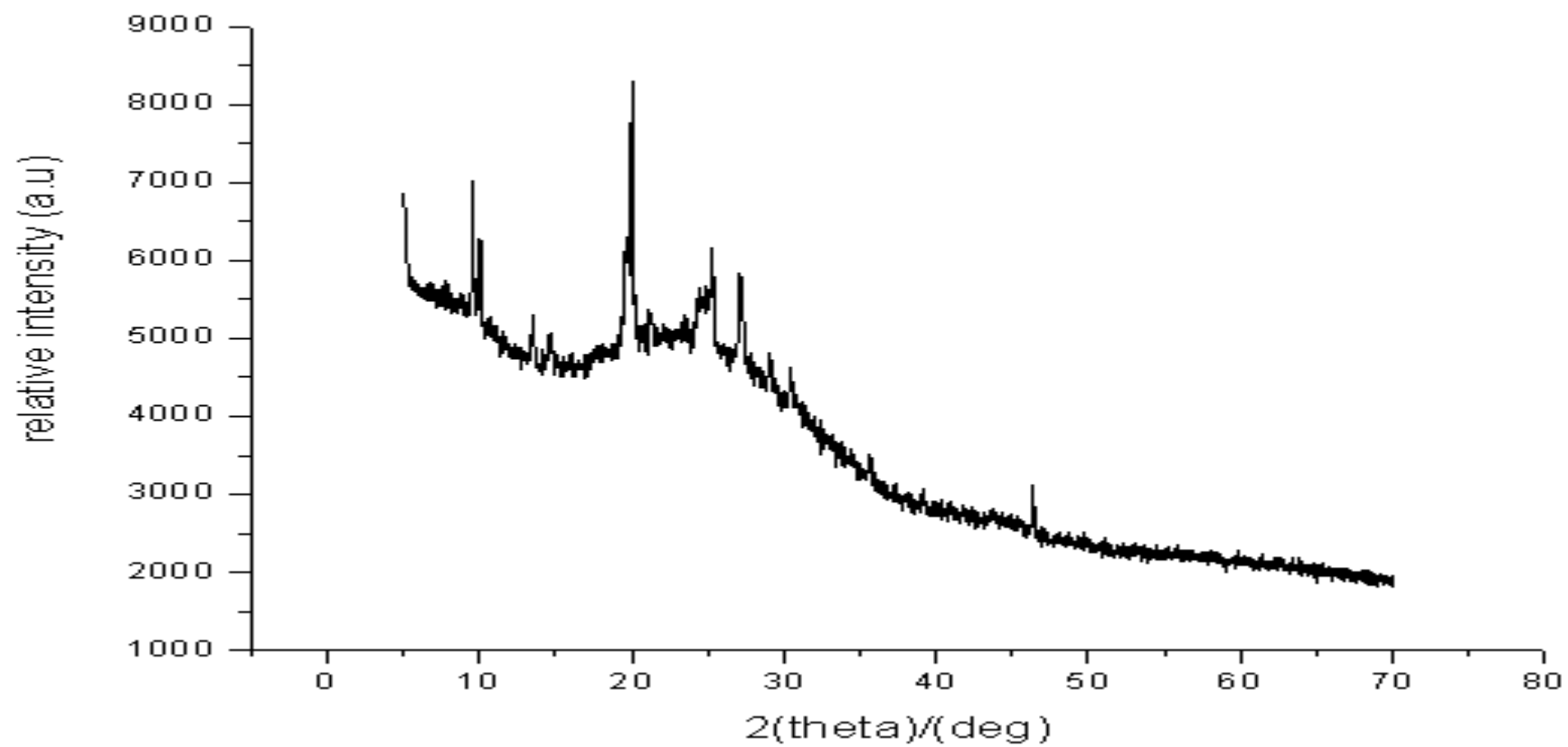


Fig.4 (12) Powder X-ray diffraction pattern of the Co(II) complex of the ligand [BCMеTB]

Table 4(11)
Powder X-ray diffraction data of the ligand [BCMeTB]

Sl. No.	2θ	θ	Sinθ	Sin ² θ	hkl	h ² +k ² +l ²	d		a. in Å°
							Calculated	Observed	
1	10.2430	5.1215	0.0892	0.00796	1 0 0	1	8.6356	8.6811	8.6370
2	20.1440	10.0720	0.1748	0.0306	2 0 0	4	4.4067	4.4112	8.8135
3	24.3984	12.1992	0.2113	0.0446	2 1 1	6	3.6455	3.6811	8.9296
4	26.4467	13.2233	0.2287	0.0523	- - -	7	3.3681	3.3856	8.9113
5	29.5194	14.7597	0.2547	0.0649	2 2 0	8	3.0243	3.0962	8.5541

Table 4(12)
Powder X-ray diffraction data of the Co(II) complex of the ligand [BCMeTB]

Sl. No.	2θ	θ	Sinθ	Sin ² θ	hkl	h ² +k ² +l ²	d		a. in Å°
							Calculated	Observed	
1	9.5874	4.7940	0.0836	0.00698	1 0 0	1	9.2141	9.2190	9.2141
2	19.9866	9.993	0.1735	0.0301	2 0 0	4	4.4397	4.4498	8.8795
3	25.2913	12.6456	0.2189	0.0479	- - -	7	3.5189	3.5629	9.3102
4	27.230	13.615	0.2353	0.0554	2 2 0	8	3.2736	3.2856	9.2594
5	46.3785	23.1892	0.3937	0.1550	3 3 2	22	1.9565	1.9761	9.9170

Naik et al.⁴, have reported the X-ray crystallographic studies of metal complexes, the results of powder x-ray diffraction data for the representative complexes are reported. The diffractograms give 10, 8 and 10 reflections for Co(II), Ni(II) and Cu(II) complexes respectively at 5-60° (2θ) with maxima at 2θ = 21.50° for Co(II), 2θ = 38.50° for Ni(II) and 2θ = 22.10° for Cu(II) complexes which correspond to d = 4.1392Å^o, 2.32Å^o and 4.021Å^o respectively all the main peaks have been indexed and their sin²θ values compared with the calculated ones. Comparison of the values reveals that there is good agreement between calculated and observed values of sin²θ. The unit cell has been calculated by the trial and error method.

Mohan et al.⁵ have studied the molecular structure of nickel (II) complex by X-ray powder diffraction pattern and data are presented. The X-ray diffraction pattern of the complex indicates the high crystallinity of the complex. The X-ray diffractogram recorded 19 reflections between 2θ ranging from 10° to 52° with maxima at 2θ = 12.0013 which corresponds to interplanar distance d = 7.3682Å^o. The main peaks have been indexed for the Sin²θ and 2θ values obtained has been compared with the calculated values. A comparison of these values revealed good agreement between calculated and observed values of Sin²θ and 2θ.

Gavali et al.⁶ have reported the powder X-ray diffraction pattern of the ligand HSTACB and its Cd(II) complex. The diffraction of HSTACB ligand consist of nine reflections between 10-80° (2θ) with maxima reflection at 2θ = 18.26 which corresponds to d= 4.8351Å^o. The diffractogram of the coordination Cd(II) complex records ten reflections between 10-80° (2θ) with maxima reflection at 2θ = 14.84 which corresponds to d= 5.9646Å^o. The X-ray pattern have been indexed by trial and error method keeping in mind the characteristic of the various symmetry system, till a good fit was obtained between the observed and indexed data. The values for HSTACB and Cd₂[HSTACB]₂Cl₂ are good fit for tetragonal system to give lattice constant that have been found to be the tetragonal type.

Zhu et al.⁷ have reported the X-ray powder lines observed for the Co(II) complex have showed satisfactorily on the basis of a unit-lattice for metal complexes. The cell parameters have been calculated by the equations⁸⁻¹¹.

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