*Available online at* www.scholarsresearchlibrary.com

Scholars Research Library

**Archives of Physics Research, 2011, 2 (2): 45-49**
*(http://scholarsresearchlibrary.com/archive.html)*

Archives of Physics Research
Scholars Research Library
**ISSN 0976-0970**
**CODEN (USA): APRRC7**

# Growth and Characterization of Ammonium Nickel Sulphate Grown by Gel Technique

**S.J.Baviskar[1], K.D.Girase[2], D.S.Bhavsar*[3]**

[1]*Dr.A.G.D.Bendale Mahila Mahavidyalaya, Jalgaon 425 003 India*
[2]*S.V.S.'s Arts and Science College, Dondaicha, 425408 India*
[3]*P.G.and Research Departments of Physics, Pratap College, Amalner 425 401 India*

---

## ABSTRACT

*Ammonium Nickel Sulphate successfully grown by simple gel technique. Then these grown crystals were characterized by XRD, IR and Thermal analysis i.e.TGA and DTA. XRD reveals that the parameters of the gel grown crystals are mostly matching with the ASTM data of the Ammonium Nickel Sulphate. IR confirms that there is strong interaction between free water molecules. While the thermal analysis indicates that the water molecules are present in the Ammonium Nickel Sulphate.*

**Keywords** gel technique, XRD, IR, TGA and DTA

---

## INTRODUCTION

Importance of gel growth can be visualized by the fact that a number of researchers have started growing crystals of technological potential. By suitable choice of reagents and their concentrations Henisch (1) was able to grow single crystals of number of substances. Murphy et al[2] used sodium sulphate as a source of sulphur for growth of lead sulphide (2). Mixed and doped crystals can also grown by the gel method by Dennis and Henisch (3). The present paper describes the growth of ammonium nickel sulphate.

## MATERIALS AND METHODS

15 gm of powder of sodium metasillicate dissolved in 250cc double distilled water through constant stirring. The mixture was then filtered by whatmann filter paper and kept into an airtight coloured bottle to avoid dust from atmosphere. Sodium metasilicate solution (sp. gr. 1.04) was used for preparation of gel. The gel was prepared from an aqueous solution of sodium

metasilicate by acidification. The vessel was corning glass tube of 2.5 cm diameter 25 cm height. 4.5 CC of $H_2SO_4$ was taken in a small beaker in which sodium metasilicate solution was added drop by drop till pH value was 4.36. While adding drops the mixture was continuously stirred by using magnetic stirrer. Then 5CC $NiSO_4$ of concentration 2M was incorporated in the above mixture. This procedure avoids excessive local ion concentration which otherwise causes premature local gelling and makes the final medium inhomogeneous and turbid. The mixture was then transferred to a test tube. The mouth of test tube was covered with cotton to avoid dust from entering into the glass tube. The solution was faint green and transparent, initially, but with lapse of time its color slightly changes. The set gel becomes slightly dark green. After aging supernatant ammonium sulphate was added slowly along the wall of the tube, so that surface of the gel was not damaged. After 8 days the crystals was taken out from test tube and cleaned for further experimentation.

X-ray diffraction studies of gel grown ammonium nickel sulphate crystal was carried on Minixlex Ragaku X-ray diffractometer available at National Chemical Laboratory, Pune. In the present study IR spectra of ammonium nickel sulphate sample was recorded using Perkin elemer FT-IR spectrophotometer at National Chemical Laboratory, Pune. The sample of ammonium nickel sulphate was analyzed by using perkin Elemer, diamond TG/DTA instrument available at National chemical Laboratory, Pune. Recrystallized alumina sample holder were used and the heating rate of $10^0$C per minue. The thermogram were recorded in the temperature range from $30^0$C to $1000^0$C.The experiment was carried out under a flow rate of 60ml per minute of nitrogen atmosphere.

## RESULTS AND DISCUSSION

### *3.1 XRD*

From x-ray diffractogram (h, k, l) values are calculated by computer programming POWD (integrative powder diffraction and indexing programming). The diffraction data matched with JCPDS data for ammonium nickel sulphate crystals. These parameters satisfy the conditions for monoclinic system i.e. $a \neq b \neq c$, and $\beta \neq 90^0$. From X-ray analysis, it may be concluded that the grown crystals of ammonium nickel sulphate have monoclinic system. The lattice parameters obtained in the present study are compared and these values matched with the reported lattice parameters.

### *3.2 IR*

According to Nakamoto and Ferraro $\nu_{OH}$ in free water molecules appears around 3600 to 3500 $cm^{-1}$, but in the present case the broad and strong band appeared at 2923 $cm^{-1}$, which is slightly lower than the expected. This may be possibly due to strong interaction between free water molecules. Another band, appearing at 1456$cm^{-1}$ may be assigned to δOH and also supports presence of free water molecules in crystal lattice as suggested by Lucchesi et al (4). IR also shows a strong band at 1152 $cm^{-1}$ indicating the presence of $SO_4^{--}$ ion in the crystal structure as predicted by Barracough et al (5).

### *3.3 Thermal Analysis*

Percentage of weight loss in the different stage of decomposition ammonium nickel sulphate crystals are observed as mentioned in the table. Thermal study confirms that crystal grown

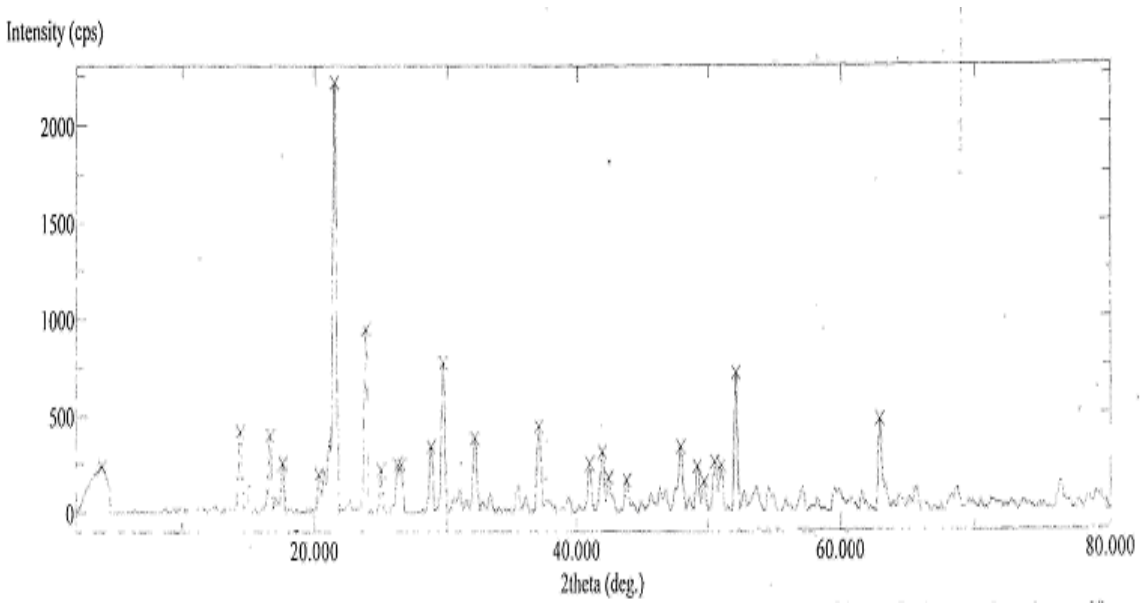
contain six water molecules in the unit cell. Hence ammonium nickel sulphate crystals are hydrated as expected. The crystals are ammonium nickel sulphate hexahydrate confirmed. The presence of metal (Ni) is confirmed from the TGA analysis. From thermogram it was concluded that the gown crystals are thermally unstable beyond $64.53^0$C and decompose into $NiSO_4$ through four stages. The residual $NiSO_4$ confirm the presence of nickel in grown crystals.

**T.G.A. data of ammonium nickel sulphate single crystal $(NH_4)_2SO_4$ $NiSO_4.6H_2O$**

| | Temperature range | % weight loss | Probable losses | Composition of the residue |
|---|---|---|---|---|
| I | 64-265$^0$C | 26.905 | $5H_2O$ | $(NH_4)_2SO_4$ $NiSO_4.H_2O$ |
| II | 265-407$^0$C | 5.626 | $H_2O$ | $(NH_4)_2SO_4$ $NiSO_4$ |
| III | 408-576$^0$C | 16.240 | 40.6% of $(NH_4)_2SO_4$ | $(NH_4)_2SO_4$ $NiSO_4$ |
| IV | 712-929$^0$C | 22.849 | 57.12% of $(NH_4)_2SO_4$ | $NiSO_4$ |

**D.T.A. data**

| Peak recorded $^0$C | Nature | Peak height mW | Onset $^0$C | Area (mJ) | ΔH(J/g) |
|---|---|---|---|---|---|
| 171.83 | Endothemic | -112.2338 | 125.52 | 41107.027 | 1007.2658 |
| 296.59 | Endothemic | -14.4878 | 279.19 | 41179.137 | 102.4035 |
| 456.84 | Endothemic | -35.1823 | 413.12 | 11323.703 | 277.4703 |
| 830.28 | Endothemic | -28.3087 | 781.20 | 8173.642 | 200.2828 |




F X-ray

**Fig.1: Diffractogram for ammonium nickel sulphate**

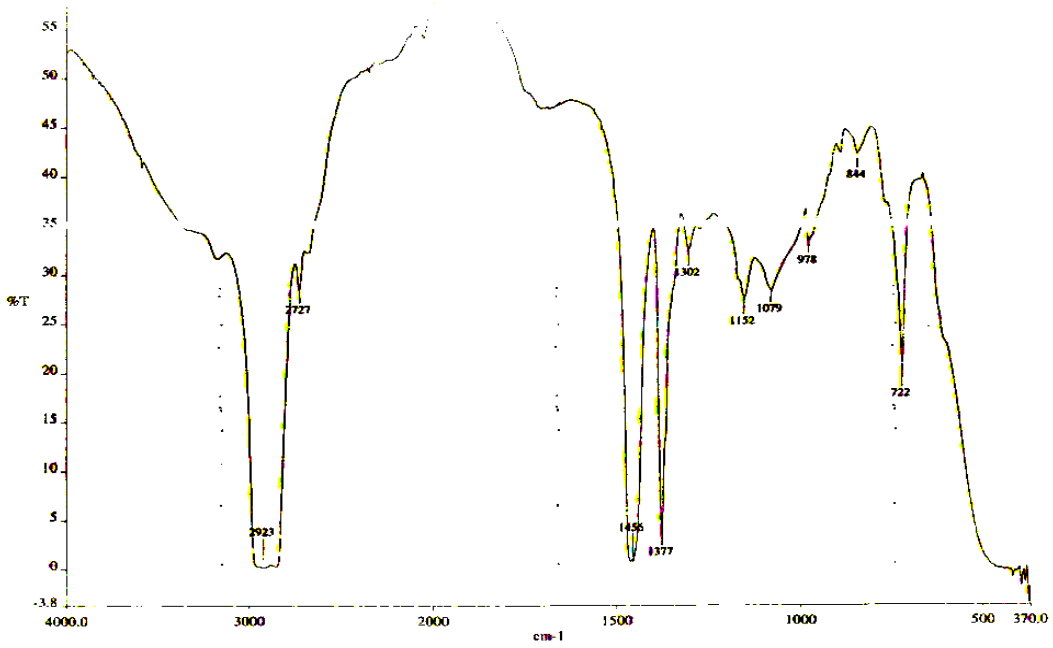



**Fig. 2: IR spectra for ammonium nickel sulphate**

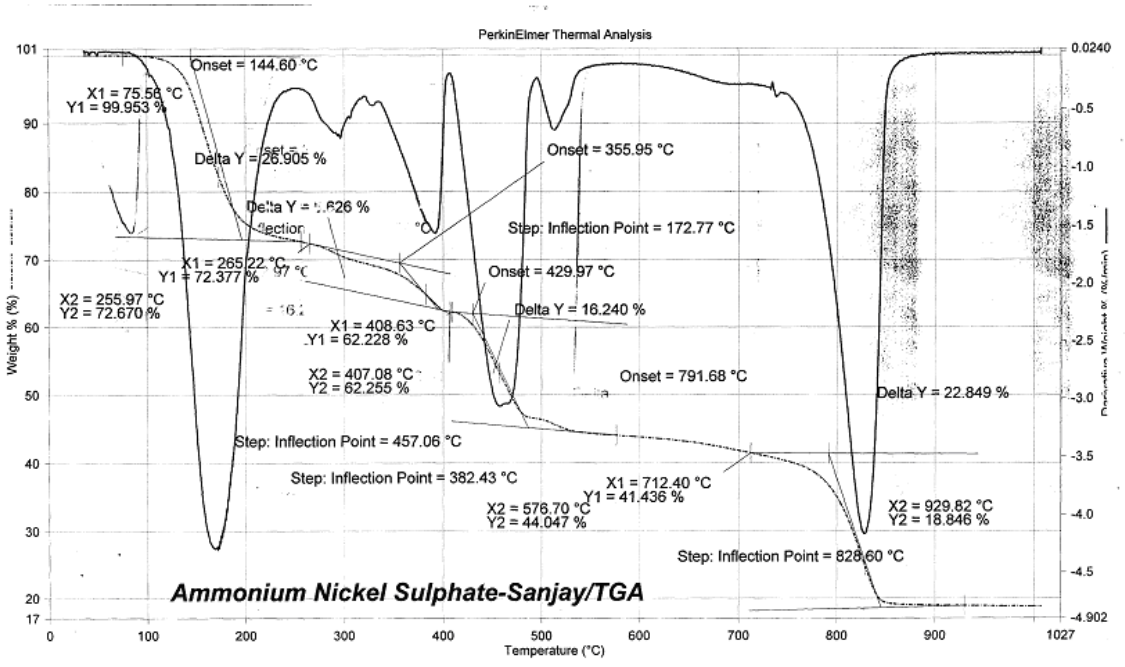
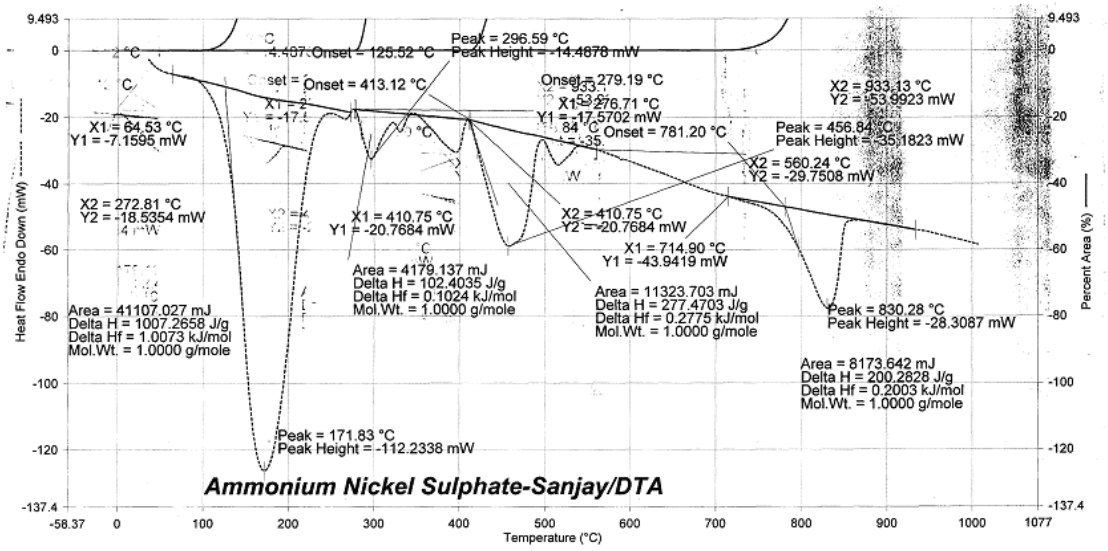



**Fig.3: TGA curve for Ammonium Nickel Sulphate**




TGA

**Fig.4: Curve for Ammonium Nickel Sulphate**

## CONCLUSIONS

1. The X.R.D.shows that grown crystal is ammonium nickel sulphate.
2. The parameter of gel grown ammonium nickel sulphate matches its J.C.P.D.S.data.
3. T.G.A.confirms six water molecule in ammonium nickel sulphate.
4. I.R . confirms $SO_4^{--}$ the present in ammonium nickel sulphate.

## REFERENCES

[1] Henisch H. K. Crystals growth in gels, The Pennsylvania State University Press , USA **1973**.
[2] Murphy J. C., Bohanday J., *J. Bul. Ame. Physics Soci.* 12, 327,**1967**
[3] Dennies J., Henisch H. K., *J. Elec. Chem. Soci.* 114, 263, **1967**
[4] Lucchesi P.J., Glassom W. A., *J. Ame. Chem. Soci.* 78, 1347, **1956**
[5] Barracough C. G., Tabe M.L., *J. Chem. Soci.*, 1993, **1961**