International Journal of Engineering, Science and Mathematics
Vol. 5 Issue 4, December 2016,
ISSN: 2320-0294 Impact Factor: 6.765
Journal Homepage: http://www.ijesm.co.in, Email: ijesmj@gmail.com
Double-Blind Peer Reviewed Refereed Open Access International Journal - Included in the International Serial Directories Indexed & Listed at: Ulrich's Periodicals Directory ©, U.S.A., Open J-Gage as well as in Cabell's Directories of Publishing Opportunities, U.S.A

Characterization of the Complex of Hepta Sulfur Imide (S_7NH) with Si (IV) By XRD Pattern

[1]U.K. Tripathi*; [1]Devendra Kumar and [2]S.P.S. Jadon

[1]Department of Chemistry, JantaMahavidyalayaAjitmal, Auraiya-206121, U.P., India

[2]S.V. College Aligarh-202001, U.P., India

Abstract :

The complex of hepta sulfur imide (S_7NH) with Si(IV) obtained by refluxing S_7NH with silicon tetra chloride, is found to have triclinic packing of it's unit cell as the values of axial distances and axial angles are found different, viz. (a#b#c) and ($\alpha \# \beta \# \gamma \# 90^0$); by XRD pattern.

Introduction :

Demarcay(1) in 1880 introduced the chemistry of sulphur-nitrogen compounds which are studied in detail continuously. Bruce, Robert B.; Gillespie, Ronald J.; Slim David R.(2)studied the crystal structures of $S_7N.S.N\ (CH_3)_2$ and N, N-thiobis (dicyclo hexyl amine). Bruce, Robert B. Stephan, Douglas W.; McGlinchey, Michael J.(3)studied the rearrangement of sulfur imide anions. Bergemann(4)prepared titanocene complexes containing bridging sulfur-bound medium sized S-N-ring ligands by the reaction of [(n^5-$C_5H_5)_2$ $Ti(CO)_2$] with cyclic sulfur imides S_7NH and $S_7N - CH_3$.Wang, ChihChieh et al(5)studied S_7NH by XRD to study its charge distribution. XRD crystallograph shows a = 7.842A^o. b = 13.115A^o and c= 7.6219A^o for S_7NH.

Bergemann, K. and co-workers[6] prepared large sulfur imides S_nNH, n=8,9,11 and studied the XRD crystallographs of S_8NH and S_9NH. U.K. Tripathi; S.P.S. Jadon[7,8] studied the XRD pattern of complex of S_7NH with tin (II) and tin (IV). In the present work, XRD-pattern of complex of S_7NH with silicon (IV) is studied which shows triclinic packing of unit cell of the crystal of the complex of S_7NH with $SiCl_4$.

Experimental :

During preparation of the complex of the ligand (S_7NH) with $SiCl_4$,doubly distilled solved and B.D.H.; Analar; S. Merk chemicals are used. The ligand (S_7NH) is prepared by passing ammonia into sulphur mono chloride dissolved in carbon tetra chloride (1:10)[8]. The solution of the ligand (S_7NH) is DMF (One gram per fifty milli liters) is mixed with the solution of silicon tetra chloride prepared in DMF (1 gm/10ml). After refluxing both solutions for 24 hours, the solid mass complex of $SiCl_4$ with S_7NH obtained is separated, washed and dried in vacuum desiccator.

From X-ray diffraction of the complex (figure-1) recorded in 2θ range from 5^0 to 70^0 (Table-1), the values of axial distances :$a_o = 10.7614A^0$,

$b_o = 10.2606A^0$ and $c_o = 3.9493A^0$; axial angels : $\alpha = 93.46^0$, $\beta = 107.78^0$ and

$\gamma = 158.39^0$ are in accordance with$a_o \# b_o \# c_o$ and $\alpha \# \beta \# \gamma \# 90^0$.

International Journal of Engineering, Science and Mathematics
Vol. 5 Issue 4, December 2016,
ISSN: 2320-0294 Impact Factor: 6.765
Journal Homepage: http://www.ijesm.co.in, Email: ijesmj@gmail.com **Double-Blind Peer Reviewed Refereed Open Access International Journal - Included in the International Serial Directories Indexed & Listed at: Ulrich's Periodicals Directory ©, U.S.A., Open J-Gage as well as in Cabell's Directories of Publishing Opportunities, U.S.A**

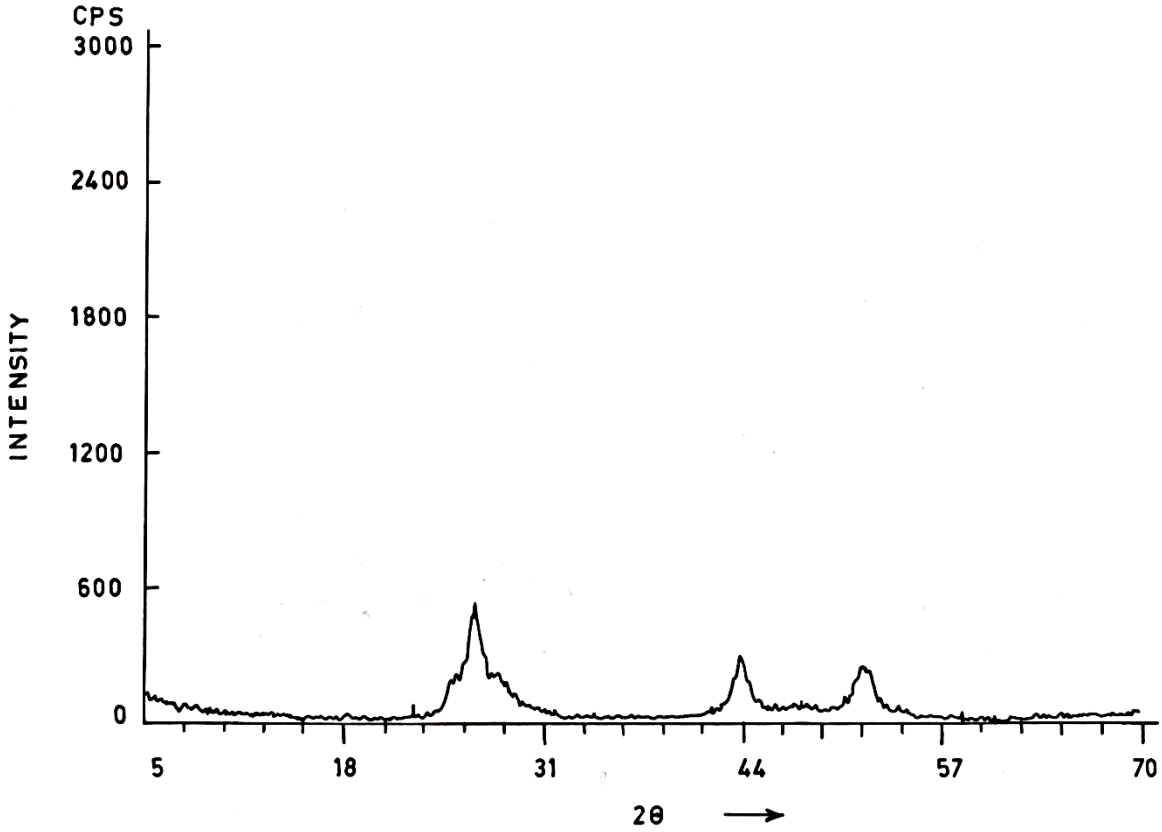

Figure-1 : XRD of the complex of hepta sulfur imide (S_7NH) with $SiCl_4$

International Journal of Engineering, Science and Mathematics
Vol. 5 Issue 4, December 2016,
ISSN: 2320-0294 Impact Factor: 6.765
Journal Homepage: http://www.ijesm.co.in, Email: ijesmj@gmail.com
Double-Blind Peer Reviewed Refereed Open Access International Journal - Included in the International Serial Directories Indexed & Listed at: Ulrich's Periodicals Directory ©, U.S.A., Open J-Gage as well as in Cabell's Directories of Publishing Opportunities, U.S.A

Table -1

XRD Pattern of the complex of S_7NH with $SiCl_4$.

S.No.	2θ	$Sin^2\theta$	Q ($h^2+k^2+l^2$)	hkl	d(A^0)
1	24.84^0	0.04626	9(0.0051)	300	3.5813
2	26.12^0	0.05106	10(0.0051)	310	3.4086
3	28.04^0	0.05869	11(0.0053)	311	3.1794
4	43.4^0	0.13671	27(0.0051)	511	2.0832
5	47.24^0	0.16054	30(0.0051)	521	1.9225
6	47.88	0.16466	33(0.0050)	522	1.8982
7	48.52	0.16882	34(0.00498)	530	1.8747
8	51.08	0.18588	36(0.00516)	600	1.7865
9	51.72	0.19025	37(0.0051)	6 1 0	1.7660
10	52.36	0.19465	38(0.0051)	6 1 1	1.7458

Discussion :

To elucidate the geometrical structure of the complex of hepta sulfur imide (S_7NH) with silicon tetra chloride (SiCl4), it's XRD is interpretated and valus of $Sin^2\theta$, miller indices (hkl) and d (distance between the planes in a crystal) is calculated from which the values of axial distances and axial angles are determined. The values of axial distances found are not equal to each other ($a_o \# b_o \# c_o$). Similarly the values of axial angles are in accordance to $\alpha \# \beta \# \gamma$

90^0, inferring that thecrystal of the complex of S_7NH with Si (IV) possess triclinic geometrical structure.

Conclusion :

From the above results of axial distances and axial angles [(a#b#c) and (α # β # γ #90^0)], it is clear that the geometry of the crystal of the complex of S_7NH with $SiCl_4$ is Triclinic.

Acknowledgements

Authorsexpress thanks to DU for providing instrumental facilities.

References :

1. Demarcy, E., Campt, Rend., 91, 854 (1880).
2. Bruce, Robert B.; Gillespie, Ronold J.; Slim, David R. : Can. J. Chem. 56(23), 2927-33 (Eng.) (1978).
3. Bruce, Robert B.; Stephan, Douglas W.; McGlinchey, Michael J. :Inorg. Chim. Acta, 53(I) L19-L20 (Eng.) (1981).
4. Bergemann, K.; Kustos, Monika; Krueger, Per; Steudil, Ralf : Angew. Chem. Int. Ed. Engl. (1995) 34(12, 1330-1 (Eng).
5. Wang, ChihChieh; Hong, Ying Ying; Ueng, Chun. Her; Wang Yu : J. Chem. Soc., Dalton Trans. 1992, (23), 3331-6 (Eng).
6. Bergemann, K.; Buschmann, J.; Luger, Peter; Angew, Chem. Int. Ed. Engl. 1996, 35(21), 2537-39 (Eng) VCH.
7. U.K. Tripathi; S.P.S. Jadon; Asian J. of chemistry, 16, 3-4, 1963-1965, (2004).
8. U.K. Tripathi; S.C. Tripathi; S.P.S. Jadon : Asian J. of chemistry 17, 2, 1221-1225, (2005).