

Table 1 Thermodynamic properties of NH_4HSO_4 , RbHSO_4 , LiNH_4SO_4 and LiCsSO_4 crystals

Crystal	T_c (°K)	$\Delta\beta^*$ $10^{-6}\cdot\text{K}^{-1}$	ΔC_p J. mol $^{-1}\cdot\text{K}$	dT_c/dP (°K/Kbar)	
				Experiment	Ehrenfest relation
NH_4HSO_4	270.9	80.5	12.2 ¹²	14 ¹³	11.3
RbHSO_4	264.5	63	9.5 ¹²	12 ⁸	11.1
LiNH_4SO_4	459.6	600 ⁷	197.5 ¹⁴	9.8 ⁷	8.8
LiCsSO_4	202.07	-30 ²	43 ²	-1.25	-1.0

Note : Numbers in superscript denote references.

* $\Delta\beta$: Change in volume thermal expansion coefficient across the transition.

* ΔC_p : Change in specific heat across the transition.

1. Kruglik, A. I., Simonov, M. A., Zhelezin, E. P. and Belov, N. V., *Sov. Phys. Dokl.*, 1979, **24**, 596.
2. Aleksandrov, K. S., Zhrebtsova, L. I., Iskornev, I. M., Kruglik, A. I., Rozanov, O. V. and Flerov, I. N., *Sov. Phys. Solid State*, 1980, **22**, 2150.
3. Delfino, M., Loiacano, G. M., Smith, W. A., Shaulov, A., Tsuo, Y. H. and Bell, M. I., *J. Solid State Chem.*, 1980, **31**, 131.
4. Raghunatha Chary, B., *Ph. D. Thesis, Indian Institute of Science, Bangalore*, 1985.
5. Raghunatha Chary, B., Bhat, H. L. and Narayanan, P. S., *Proc. 3rd Natl. seminar on ferroelectrics and dielectrics*.
6. Anistratov, A. T., Zamkov, A. V., Kot, L. A., Stdovitskaya, I. N. and Shabanova, L. A., *Ferroelectrics*, 1983, **48**, 103.
7. Shimizu, H., Oguri, A., Yasuda, N. and Fujimoto, S., *J. Phys. Soc. Jpn.*, 1978, **45**, 565.
8. Gesi, K. and Ozawa, K., *J. Phys. Soc. Jpn.*, 1975, **38**, 459.
9. Tamaki, A. and Fujimura, T., *J. Phys. Soc. Jpn.*, 1983, **52**, 2922.
10. Iskornev, I. M. and Flerov, I. N. *Sov. Phys. Solid State*, 1977, **19**, 605.
11. Iskornev, I. M. and Flerov, I. N. *Sov. Phys. Solid State*, 1978, **20**, 1531.
12. Aleksandrov, K. S., Anistratov, A. T., Blat, D. Kh., Zhrebtsova, L. I., Zinenko, V. I., Iskornev, I. M., Melnikova, S. V. and Flerov, I. N., *Ferroelectrics*, 1976, **12**, 191.
13. Polandov, I. N., Mylov, V. P. and Strukov, B. A., *Sov. Phys. Solid State*, 1969, **10**, 1754.
14. Loicono, G. M., Delfino, M., Smith, W. A., Bell, M. I., Shaulov, A. and Tsuo, Y. H.,

Ferroelectrics, 1980, **23**, 89.

15. Samara, G.A., Sakudo, T. and Yoshimitsu, K., *Phys. Rev. Lett.*, 1975, **35**, 1967.
16. Samara, G. A., *Comm. Solid State Phys.*, 1977, **8**, 13.
17. Wadhawan, V. K., *Phase transitions*, 1982, **3**, 50.

SPECTROPHOTOMETRIC DETERMINATION OF BERYLLIUM(II) WITH 1-(2-THIAZOLYLAZO)-2-NAPHTHOL IN PRESENCE OF TRITON X-100

M. C. ESHWAR and G. V. RATHAIAH
Department of Chemistry, Indian Institute of Technology, Bombay 400 076, India.

EVEN though pyridylazo and thiazolylazo dyes find wide application^{1,2} as analytical reagents, no thiazolylazo derivative has been employed for the determination of beryllium(II). It has been found that the reaction between 1-(2-thiazolylazo)-2-naphthol, TAN, and beryllium(II) is suitable for spectrophotometric determination of beryllium.

Absorbance measurements were made on Spectromom 204 UV-visible spectrophotometer and absorption spectra recorded on Superscan 3 UV-visible spectrophotometer. A Cambridge pH meter was employed for pH measurements. Stock solution of beryllium(II), prepared from beryllium nitrate tetrahydrate (E Merck), was standardized gravimetrically³. A 0.1% (w/v) methanolic solution

of TAN was prepared from 1-(2-thiazolylazo)-2-naphthol (Fluka) and also aqueous solution of Triton X-100 (Fluka). Buffer solution (pH 6.6) was prepared from 0.5 M solutions of hexamine and hydrochloric acid (A.R.).

The optimum pH for the reaction between beryllium(II) and TAN is 6.3 to 6.9 and the reaction was studied at pH 6.6, maintained by hexamine-HCl buffer. The sparingly soluble complex dissolves in polar organic solvents requiring at least 80% (v/v) of the solvent, and maximum absorbance is noticed in acetone. The complex is however found to be water soluble in the presence of non-ionic surfactant, Triton X-100. Although the complex has equal sensitivity in both media, surfactant medium has been preferred for the determination to avoid the use of high concentration of organic solvent. At least 2.5 ml of 10% Triton X-100 solution is required for solubilization and constant maximum absorbance is obtained in the presence of 2.5 to 4.5 ml of surfactant and 4 to 7 ml of 0.1% methanolic solution of TAN. The complex exhibits maximum absorbance at 547 nm in 80% (v/v) acetone and 555 nm in surfactant medium. The instantaneously formed complex is stable for 24 hr in acetone but starts precipitating after 2 hr in surfactant medium.

The 1 : 2 complex obeys Beer's law over the concentration range 0.02 to 0.34 ppm of beryllium, has molar absorptivity $2.25 \times 10^4 \text{ l mol}^{-1} \text{ cm}^{-1}$, Sandell sensitivity 0.4 ng cm^{-2} and coefficient of variation $\pm 0.76\%$. Tolerance limits of various ions in determination of $4 \mu\text{g}$ of beryllium(II) in 25 ml are (in μg): Cl^- , Br^- , I^- , urea, thiourea 20000; $\text{S}_2\text{O}_3^{2-}$ 10000; ascorbate 7500; SO_4^{2-} 5000; $\text{NH}_2\text{OH.HCl}$ 4000; Mo(VI), W(VI), Pt(IV) 2000; phthalate, Au(III) 1000; succinate, Al(III) 500; $\text{N}_2\text{H}_6\text{SO}_4$ 400; CN^- 300; V(V), Th(IV), Cr(III), Ag(I) 200; oxalate 150; Ga(III) 100; In(III), Hg(II) 40; Sb(III), Bi(III), Sn(II) 20; PO_4^{3-} , malonate, Pb(II) 10; F^- , U(VI), Tl(III), Pd(II) 5. Alkali and alkaline earth metal ions, CO_3^{2-} , SCN^- did not interfere even in large amounts. EDTA and citrate gave negative interference, whereas Fe(II, III), Mn(II), Co(II), Ni(II), Cu(II) and Zn(II) gave positive interference.

An aliquot of the solution containing $\leq 8.5 \mu\text{g}$ of beryllium(II) is taken, added 5 ml of 0.5 M hexamine-HCl buffer, 4 ml of 10% Triton X-100, 6 ml of 0.1% methanolic TAN, the contents diluted to 25 ml with distilled water and absorbance measured at 555 nm against a reagent blank prepared under identical conditions.

Beryl (0.5 g) was fused with sodium carbonate and brought into solution⁴. An aliquot of this solution was taken, beryllium separated by reversed phase extraction chromatography⁵ and determined as given earlier. Duplicate analysis gave the beryllium content as 4.68 and 4.69% against the theoretical amount of 4.71%.

One of the authors (GVR) thanks CSIR, New Delhi for the award of a fellowship.

27 May 1985

1. Anderson, R. G. and Nickless, G., *Analyst*, 1967, **92**, 207.
2. Havard R. Hovind, *Analyst*, 1975, **100**, 769.
3. Vogel, A. I., *A textbook of quantitative inorganic analysis*, Longman, London, 1975, p. 518.
4. Shankar Das, M. and Athavale, V. T., *Anal. Chim. Acta*, 1955, **12**, 414.
5. Chhaya Sharma and Khopkar, S. M., *Anal. Chim. Acta*, 1985, **167**, 403.

EFFECTS OF CODEINE ON RAT INTESTINE

N. SEKAR, S. WILLIAM and
S. GOVINDASAMY

Department of Biochemistry, University of Madras,
Madras 600 025, India.

CODEINE, first reported by Robiquet 1832 (see ref. 1), is an alkaloid of opium, obtained from the milky exudate of the incised unripe seed capsules of the poppy plant, *Papaver somniferum*. This belongs to the opiate group such as morphine and codeine which are used principally as analgesics, to induce sleep in the presence of pain, to check diarrhoea and to suppress cough. It has adverse effects like euphoria, drowsiness, decreased gastrointestinal motility, reduced propulsive movement and increased constipation².

The effects and mechanism of action of opiates on the gastrointestinal nonpropulsion have been reported earlier³⁻⁶. The constipating effects of opiates have been investigated in several mammalian species⁷⁻⁹. In rats a delay in transit time in small intestine induced by opiate has been associated with decreased contractile activity^{10,11}.