MOSSBAUER STUDY OF THE IRON (III) COMPLEXES OF PYROPHOSPHORIC ACID

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ABSTRACT

The complexes of iron (III) with pyrophosphate toth in acidic ard alkaline medium were isolated. Their chemical formulae were established using chemical analysis data. The Mössbauer spectra of the two complexes, i.e., H (Fe (H₂P₂O₇)₂ (H₂O)₂I.4H₂O and Na₅ [Fe (P₂O₇)₂ (H₂O)].3H₂O gave the same values of Isomer shift. This fact go to show that the electron density in both the complexes is roughly the same but the difference in quadrupole splitting of the two type of complexes predicts that the symmetry around the ferric ion in case of complex formed in acidic medium is more distorted than in the alkaline medium.

Introduction

on metal pyrophos-I ITERATURE survey phates 1-8 would reveal that almost all the investigations have been carried out with the products obtained by the reaction of sodium pyrophosphate with the metal salts. In view of the fact that sodium pyrophosphate solution cannot exist in a stable form⁹⁻¹⁰, the results achieved by such studies are likely to be less reliable. However, if studies on metal pyrophosphates are carried out by using pyrophosphoric acid, more meaningful data can be achieved. Also, no Mössbauer spectral studies either in the case of pyrophosphoric acid or sodium pyrophosphate complexes have been reported so far. In the present communication the Mossbauer study of the complexes of iron (III) with pyrophosphoric acid as well as with sodium pyrophosphate is presented.

EXPERIMENTAL

Reagents and Solutions.—All the reagents employed were of A. R. quality. The pyrophosphoric acid and sodium pyrophosphate used were of MERCK Products. All the solutions were made in double distilled water.

Preparation of the Complexes.—Solutions of ferric nitrate and pyrophosphoric acid (Conc. $1 \cdot 0$ M each) were mixed in the molar ratio of 1:2 and allowed to stand for about 2 hours. The precipitate obtained was filtered, washed several times with water and then dried at 90° C. The complex of iron (III) with $Na_4P_2O_7$ was prepared similarly.

The spectra of the two type of complexes were run by constant Acceleration Velocity Drive Moss-bauer Spectrometer.

RESULTS AND DISCUSSION

From the chemical analysis, the molecular formulae of the two complexes were established to be $H[Fe(H_2P_2O_7)_2(H_2O)_2].4H_2O$, and $Na_5[Fe(P_2O_7)_2(H_2O)_2].3H_2O$.

Mossbauer Study of H[Fe($H_2P_2O_7$)₂ (H_2O)₂].4 H_2O . The observed values of Isomer shift and Quadrupole splitting (Fig. 1) are 0.433 mm/sec.

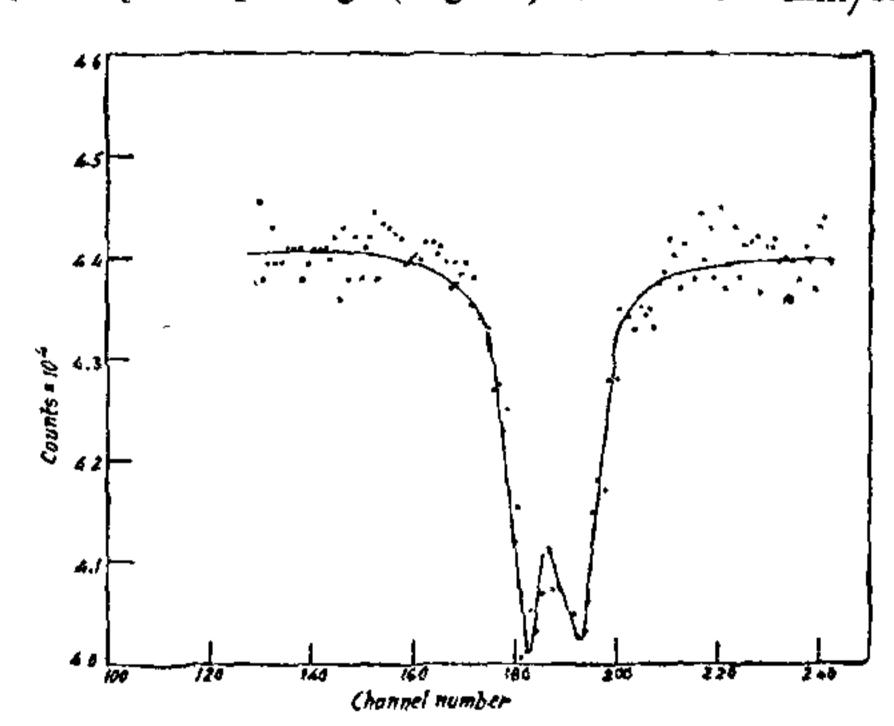


Fig. 1. Mössbauer spectrum of $H_2\{Fe(H_2P_2O_7)_2(H_2O)_2\}\cdot 4H_2O$.

(w.r. to Fe) and 0.671 mm/sec, respectively. Iron is bonded through Fe-O bonding with its ligands and the symmetry formed by the surrounding ligands is octahedral with a covalent bonding and iron exists in Fe (III) form. The values of I.S. and Q.S. observed supports the view that iron is in Fe (III) form and the symmetry around from is octahedral with a little distortion due to different ligand groups (P-O-H, O-P-O) responsible to octahedral symmetry. The infra-red studies go to show that the stretching frequencies of the P-O-II and O-P-O groups are shifted on complexation and thereby these groups are responsible for distortion. Further magnetic moment values which comes out to be 5.83 B.M., confirms that it is a high spire Te (III) complex.

Study of Na₅[Fe(P₂O₇)₂ (H₂O)₂] 3 H₂O₂—The observed values of LS, and QS, are, 3 =0.44 mm sec. (n.r. to Fe) and 4 = 0.46 mm/sec.

As discussed in the study of $H[Fe(H_1P_2O_1)_2 (H_2O_1)_2].4H_2O$, here too, iron is in Fe (III) state with octahedral surrounding and the ligand groups are bonded through covalent bonding forming $Na_3[Fe(P_2O_7)_2, (H_2O)_2].3H_2O$.

Comparison of the Spectra.—Mossbauer spectra give the same values of Isomer shift in the two cases, i.e., 0.433 mm/sec. in the case of pyrophosphoric acid complex and 0.44 mm/sec. in the sodium pyrophosphate complex. This goes to show that the electron density in both the phosphate complexes is roughly the same but the difference in quadrupole splitting in these two types of complexes shows that the symmetry around ferric ion in pyrophosphoric acid complex is more distorted than in the complex of sodium pyrophosphate.

- 1. Rogers, L. B. and Reynolds, C. A., J. Am. Chem. Soc., 1949, 71, 2081.
- 2. Subrahmanya, R. S., J. Indian Inst. Sci., 1956, 38 A. 87.
- 3. Banerjee, S and Mitra, S. K., Sci. and Cult., 1951, 16, 530.
- 4. Friksson, E. and Hogskol, K. L., Ann., 1949, 16, 39.
- 5. Watters, J. I. and Kolthoff, I. M., J. Am. Chem. Soc., 1948, 70 (23), 2455.
- 6. Vaid, J. and Ramachar, T. L., Curr. Sci. 1954, 23, 396.
- 7. Haldar, B. C., Nature, 1950, 166, 744.
- 8. Mustaev, A. K. and Yakdovenko, T. L. Issled. Khim. Redkikh Soputstuuyushchikh Elem. Akad. Nauk. King. SSR, Inst. Neurg. Fig. Khim., 1966, 66, 69339 X, (Russ.), 82.
- 9. Growther. J. P. and Westman, A. E. R., Cand. J. Chem., 1954, 32, 42.
- 10. Vanwazer, J. R., Griffith, E. J. and McCullough, J. F., J. Am. Chem. Soc., 1955, 77, 287.

CENTRAL STIMULANT ACTIVITY OF A BENZOCYCLOHEPTENE DERIVATIVE IN MICE: COMPARISON WITH (+) AMPHETAMINE

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ABSTRACT

7-Dimethylamino-5, 6, 8, 9-tetrahydro-7-phenyl-7H-ber zocyclohepter e hydrochloride has been tested for its central stimulant activity by using two models in mice. This compound ircreases spontaneous locomotor activity and antagonises reserpine-induced catalepsy and hypothermia. The results reveal that it could possibly stimulate the central nervous system like (+) amphetamine.

INTRODUCTION

PRELIMINARY studies with the compound, 7-Dimethylamino-5, 6, 8, 9-tetrahydro-7-phenyl-7H-benzocycloheptene hydrochloride** showed that it could stimulate the central nervous system in mice and hence it was decided to test this property at three dose levels in two models, (1) on spontaneous locomotor activity, (2) against reserpine-induced catalepsy and hypothermia. Results obtained in such a study provide the basis for this report.

MATERIALS AND METHODS

(a) Stimulation of Spontaneous Locomotor Activity in mice

Spontaneous locomotor activity was taken as a parameter of behavioural excitation. It was recorded by means of a conventional light beam cage^{2,3}. The light and sound-proof cabinet contained 4 identical cages, each 22 cm × 37 cm × 8 cm, crossed by 6 light beams. Five mice (18-25 g)

** Also known by the Code SYA-33118.

were placed in each cage 15 min. after subcutaneous administration of the compound/vehicle and the number of light beam interruptions taking place was recorded at 30 min. intervals for 7 hours, through an automatic counter. Experiments were repeated 6 times, giving a total of 30 mice per treatment. The mean total 'motility count for 5 mice over the recording period, and the mean, and Standard error of mean were calculated. Statistical comparisons were carried out by means of Student's 't' test.

The experiments were performed on male albino mice (NMRI Strain). The animals were kept under standard laboratory conditions (constant temperature 22 ± 1°C, humidity 60% and light on between 8 a.m. and 8 p.m.) and had no prior experience in the activity cages.

The substance was dissolved in one or two drops of glacial acetic acid and the pH was adjusted to 5-6. Control animals received the vehicle at the same pH. Three doses of the substance, 2.5, 5 and 10 mg Kg⁻¹ were studied.

For comparison purposes, (+) amphetamine sulphate was administered subcutaneously at similar

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