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SYNTHESIS AND ANALYSIS OF SODIUM TRIPOLYPHOSPHATE PEROXO SOLVATE

A. I. MARTIRYAN [1*], L. A. NERSESIAN [2]

[1] *Chair of Ecological Chemistry YSU, Armenia*
[2] *Institute of Chemical Physics NAS of RA*

A method of sodium tripolyphosphate peroxo solvate synthesis has been developed. It has been shown that the hydrogen peroxide content in peroxo solvate is 28 wt.% and the solid product exists in crystalline form.

***Keywords*:** peroxo solvate, hydrogen peroxide, sodium tripolyphosphate.

Introduction. One of the typical properties of hydrogen peroxide is the formation of peroxo solvates [1–3]. Solvation reactions is the only area in hydrogen peroxide chemistry, where H_2O_2 molecules, despite the tendency to catalytic decomposition, stay intact and change from liquid state to solid as coordinated ligands [1].

The peculiarity of hydrogen peroxide is the specific coordination of H_2O_2 molecules to the salts not as could be expected, based on the presence of two oxygen atoms composition in hydrogen peroxide, through which, like water molecules, hydrogen peroxide is achieved by other mechanism, namely via formation of hydrogen bonds of H_2O_2 with salt anions [1–4].

There are a number of studies that are aimed at obtaining water soluble peroxo solvates albeit it is mentioned that the systematic research in this area is limited [1–4]. In recent years special attention is paid to the synthesis and practical application of solid forms of hydrogen peroxide. Peroxide products can be used for cancer treatment and for oral administration in case of poisoning [5]. Certain metal peroxide compounds are used in the purification process of wastewater that contains arsenic, manganese, chromium and copper ions. Calcium and magnesium compounds of peroxide and peroxo solvate are used to remove odor, color and in neutralization of harmful chemical and biological agents in the tertiary wastewater treatment. Granular forms, as well as the suspensions of the peroxy compounds, are used for cleaning harmful gas emissions resulting from chemical industry such as sulfur dioxide and nitrogen oxides [1–3].

Solvates containing hydrogen peroxide, i.e. peroxo solvates form many inorganic salts and certain organic compounds [1–5].

* E-mail: armart@ysu.am

Many salts of phosphoric acid such as Na_3PO_4, $Na(NH_4)HPO_4$, $(NH_4)_2HPO_4$, $NH_4H_2PO_4$, $Mg(NH_4)PO_4$, $Na_4P_2O_7$, $K_4P_2O_7$, $(NH_4)_4P_2O_7$, $Ca_2P_2O_7$, $Sr_2P_2O_7$ and $Ba_2P_2O_7$, tend to form peroxo solvates. The ability of phosphates to incorporate a hydrogen peroxide grows from sodium to potassium salts, as well as in the anion row $H_2PO_4^- - HPO_4^{2-} - P_2O_7^{4-}$. As sodium phosphate peroxo solvates are mentioned $Na_2HPO_4 \cdot 2H_2O_2$, $Na_2HPO_4 \cdot 0.75\ H_2O_2$, $Na_3PO_4 \cdot 2H_2O_2$, $Na_3PO_4 \cdot 5H_2O_2$ [6].

Properties of metal phosphate peroxo solvates are poorly studied, as these compounds are rarely found in the individual state and as mixtures with hydrates they are unstable. It is known that only sodium pyrophosphate peroxo solvate has high storage stability. Within 6 months solvate loses 1% of hydrogen peroxide.

Nowadays, application areas of different metals phosphate peroxo solvates are practically unknown. It is just possible to assume that the salts of the phosphoric acid can be used in pharmaceutical industry, in the composition of specific detergents, as disinfectants, in the processing of fabrics before dyeing and as food additives. Among the salts of phosphoric acid, a great practical interest represents sodium tripolyphosphate (STTP), which is approved for the use in food industry (code E451) as food additive: a complexing agent, an acidity regulator, a thickener, a binder, flour and bread quality improver; it is also a part of special drinks for the athletes. Besides, STTP in a detergent helps to emulsify fat, reduces water hardness and therefore, it is included in the composition of many detergents for dishwashers and washing machines [7].

There are no publications about the production process of STTP peroxo solvate and its physical chemical properties. The goal of this study is to develop a method of producing STTP peroxo solvate, to determine the content of hydrogen peroxide in the peroxo solvate and to study some structural features of the obtained product.

Experimental Part. For synthesis of STTP peroxo solvate under laboratory conditions several methods were tested [8, 9]. The most appropriate one was the following: a 30 wt.% solution of hydrogen peroxide was poured into Erlenmeyer flask. The flask with solution was stirred on magnetic stirrer and cooled up to 0°*C*, then while stirring the STTP was added (the amount of the salt was considerably smaller then the amount of peroxide in the solution), incubation period of 40 *min* followed, after the mixture was poured into another flask, it was cooled up to –15°*C* and incubated at this temperature for 2 *h*. The precipitate was filtered and dried in an oven at 50°*C* in a steam of air. The yield of the product (via STTP) was 68–70%.

Hydrogen peroxide content in the obtained product was determined using permanganometric method. The obtained compounds were mixed with distilled water and the amount of hydrogen peroxide in the liquid state was determined by titration. The total amount of hydrogen peroxide in the solution was measured by the means of titration which was continued until the color of solution was stable for 30 *min*. The total amount of hydrogen peroxide in STTP peroxo solvate was three moles per one mole $Na_5P_3O_{10}$ ($Na_5P_3O_{10} \cdot 3H_2O_2$), i.e. the concentration of hydrogen peroxide in product was 28 wt.%.

An X-ray diffractometer DRON-3 was used in order to study the structural features of STTP peroxo solvate. Diffractograms are shown in Fig. 1.

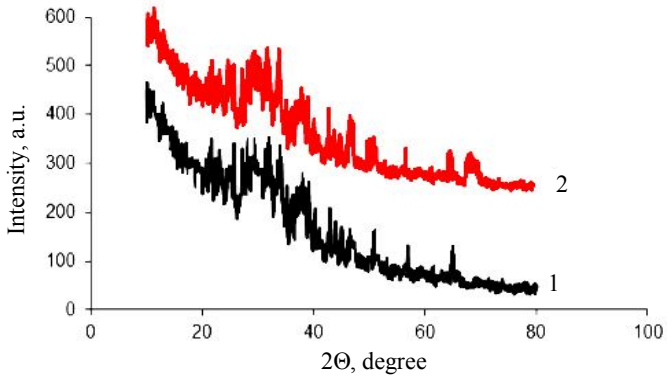

Fig. 1. The diffractograms of samples: 1 – STTP; 2 – STTP peroxo solvate.

The attained diffraction patterns show that the compound obtained by reaction of STTP and hydrogen peroxide converts into crystalline state after drying.

IR spectra of STTP and its peroxo solvate were obtained by the use of Nicolet / FTIR NEXUS spectrometer (Fig. 2).

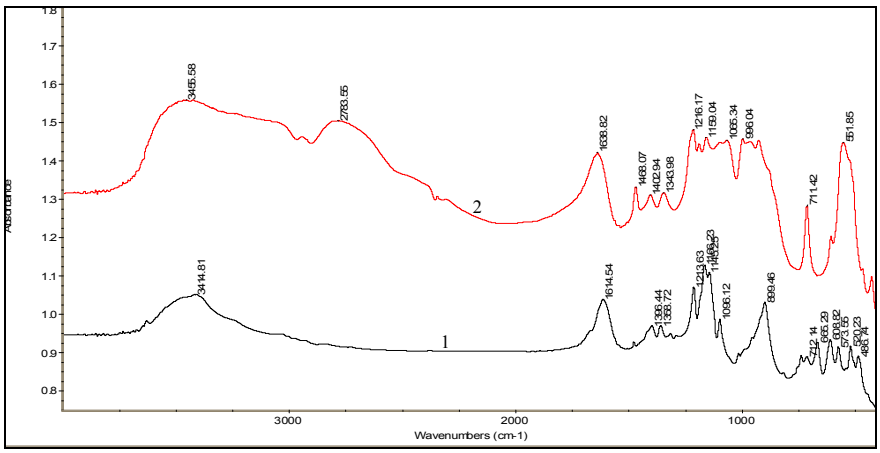

Fig. 2. FTIR spectra of STTP (1) and STTP peroxo solvate (2).

Comparative analysis of the changes in the stretching frequencies of P–O (1050–1500 cm^{-1}) and P–OH (450–970 cm^{-1}) groups in the IR spectra (Fig. 2) shows that the compound obtained by the reaction of STTP with hydrogen peroxide contains bounded hydrogen peroxide, which affirms the formation of peroxo solvate. The results of IR measurements and X-ray analysis show that in the reactions of STTP with hydrogen peroxide STTP peroxo solvates are formed.

Thus, a method of STTP peroxo solvate synthesis was developed. It has been demonstrated that the amount of hydrogen peroxide in peroxo solvate is 28 wt.% and the obtained product converts into crystalline state after drying.

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