

## PREPARATION OF PURE HYPOPHOSPHOROUS ACID

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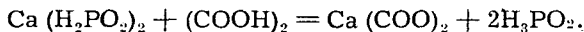
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Pure hypophosphorous acid, *i. e.* not contaminated with phosphoric or phosphorous acid is often needed in inorganic and organic chemical researches. According to JENKINS and JONES [1], the commercial 50% hypophosphorous acid always contains a few per cent of phosphoric and phosphorous acids. The process proposed by these authors to remove impurities, seems, however, to be cumbersome and, in addition, accompanied by a considerable loss.

The necessity arose in this Laboratory to prepare carefully purified hypophosphorous acid for the reductive deamination of an aromatic amino group [2], [3]. To meet this purpose, a new simpler and more comfortable method was evolved.

Since we disposed of pure calcium hypophosphite, it seemed practical to liberate hypophosphorous acid by any acid yielding a poorly soluble calcium salt. The high water solubility of calcium sulphate rendered sulphuric acid impractical for this purpose, whereas oxalic acid made possible the quantitative precipitation of calcium.

An equivalent amount of calcium hypophosphite proved to react in an aqueous solution with oxalic acid according to the following equation:



The dilute aqueous solution of hypophosphorous acid obtained was concentrated *in vacuo* in a nitrogen stream.

### Experimental

Calcium hypophosphite (340,2 g, 2 mol) was dissolved in hot distilled water (2.500 ml) and a solution of crystalline oxalic acid (hydrate) (252,14 g, 2 mol) in hot water (100 ml) was added under stirring. After allowing to cool, the white massive precipitate of calcium oxalate was filtered. The solution, in turn, was evaporated to 384 ml in a nitrogen stream *in vacuo* on a water-bath of 45° C. Titration showed a content of 52 per cent hypophosphorous acid. Dilution with water yielded the concentration required.

Based upon analytical data obtained by the JONES and SWIFT [4] method, our product contained less than 0,1 per cent phosphoric and phosphorous acid,

resp. The hypophosphorous acid solution prepared in this way gave fair yields in certain reductive deamination processes.

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#### References

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- [3] Bánfi, D., G. Benke, G. Fodor, J. Kiss *et al.*: *Magyar Kém. Foly.*, **85**, 225 (1952).
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