

Apparatus and Technique

The Electrolytic Preparation of Sodium Hyponitrite

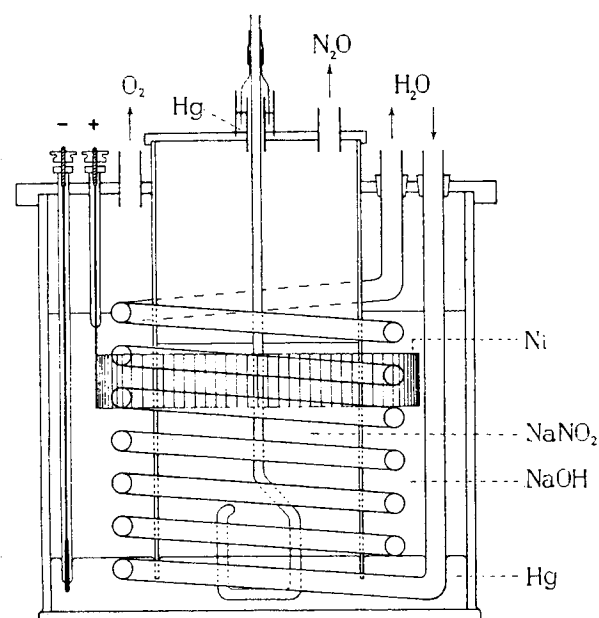
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The most frequently employed method of preparing hyponitrites is by reducing sodium nitrite with sodium amalgam. The detailed characteristics of this method are well known.¹

The electrolytic method does not seem to have attracted the deserved attention. Electrolysis of a NaNO_2 solution with a mercury layer serving as the negative electrode was first proposed by Zorn.² The method was radically modified by Abel³ but it has not been used ever since.

The electrolytic cell shown diagrammatically in the figure is a simplification of Abel's cell, which was more complicated than practically necessary. It consists of



two concentric jars, the inner of which is bottomless and dips into a mercury layer connected with the negative pole. The anode is a coiled nickel wire or gauze. Electrolysis of concentrated (almost saturated) NaOH in the outer compartment forms sodium amalgam, which by stirring passes inside and reacts with the solution of NaNO_2 . The process (electrolysis and stirring) is carried on as long as foaming (by evolution of N_2O) is observed in the inner compartment. Then the product (alkaline solution of $\text{Na}_2\text{N}_2\text{O}_2$) is withdrawn by siphoning and treated as desired.¹

Blackening of the nickel anode reveals that the concentration of the solution of NaOH has been reduced too much. If so this solution is replaced by a more concentrated one.

The diameter and the height of the outer jar are of about 20 cm. each. The cover is made of alkali-resistant plastic material.

For a current of about 4 amp., 3-4 volt. are needed. The current is used almost entirely in the formation of sodium amalgam (no H_2 is evolved).

The concentration of the original NaNO_2 solution depends on the desired concentration of the product.¹

The merits of the electrolytic method (no need of metallic sodium and the possibility of overnight work) are unquestionable, particularly if large amounts of hyponitrite are needed. The author has used the cell described over more than four years and found it quite satisfactory.

Thanks are due to Dr. Pandora Nikolaïdes and Mr. M. Pipinis for their assistance in the construction of the apparatus.

References

- 1 Polydoropoulos, C. N., *Chimika Chronika*, 1959, 24A, 147
- 2 Zorn, W., *Ber. der deut. chem. Ges.*, 1879, 12, 1509
- 3 Abel, E., Orlicek, A. & Proisl, J., *Wien. Monatsh*, 1938, 72, 1

A Convenient Method for the Removal of Iodine from Aqueous Solutions of Hydriodic Acid

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Aqueous hydriodic acid readily oxidises to iodine on standing, as is shown by the progressive yellowing of the solution. The effect is most marked in the case of concentrated solutions, but is perceptible at con-

centrations as low as 0.05M, and cannot be prevented except by the most rigorous exclusion of oxygen.¹

The removal of iodine from these partially oxidised solutions is often necessary, but is difficult because of