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NOTE

N-Chloronicotinamide as an Oxidimetric Titrant: Potentiometric Titrations of Some Typical Common Reductants in Aqueous Acetic Acid Medium

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N-Chloronicotinamide (NCN) has been used as an oxidimetric titrant in aqueous acetic acid medium. Potentiometric titrations of some typical common reductants have been performed using N-chloronicotinamide.

N-Halo compounds such as N-chlorosuccinimide (NCS)[1], N-chlorosaccharin (NCSA)[2], N-bromosaccharin (NBSA)[3] and N-bromophthalimide (NBP)[4] have already been used as potentiometric oxidimetric titrants in aqueous acetic acid medium. The utility of N-chloronicotinamide (NCN) as an oxidimetric titrant has not been explored so far, and hence the present investigation aims to use N-chloronicotinamide as a titrant in the potentiometric determination of some common reductants.

A digital potentiometer (Equiptronics) with a platinum-saturated calomel electrode assembly and magnetic stirrer were used for potentiometric titrations.

NCN was prepared and characterised by literature method[5] and its purity was checked by iodometric titration. For analytical work fresh solutions of NCN were prepared in anhydrous acetic acid (E. Merck). Standard solutions of reductants were prepared in acetic acid-water medium using 2N HCl.

Potentiometric Technique: The oxidant solution was taken in a 100 mL beaker (0.1 g of NCN dissolved in 20 mL of glacial acetic acid) in which a Pt-SCE assembly was set up. The reductant solution was taken in a microburette and was added to the titrant solution in 0.2 mL additions. The experimental solution was stirred automatically using magnetic stirrer and the change in emf was followed. The titrations were continued until there was no significant change in potential on further addition of the titrant. The equivalence points were located by graphical method[6]. The mass of the reductant dissolved determined by potentiometric method was compared with the mass of the reductant weighed.

The following eight reductants have been estimated using NCN in different concentrations:

(a) Thiourea, (b) Ascorbic acid, (c) Succinic acid, (d) β-Naphthol, (e) Anthranilic acid, (f) Mandelic acid, (g) Semicarbazide, (h) Iodide (I^-).

The results of potentiometric method for eight reductants are given in Table-1.

TABLE-1

Reductant	Range studied (mmol)	Standard deviation (μmol)	Average error (%)
Thiourea	0.02–0.30	0.5	±0.42
Ascorbic acid	0.01–0.50	0.8	±0.51
Succinic acid	0.09–0.17	1.0	±0.09
β-Naphthol	0.03–0.10	1.2	±0.18
Anthranilic acid	0.04–0.18	1.6	±0.24
Semicarbazide	0.03–0.12	0.9	±0.13
Mandelic acid	0.02–0.08	2.5	±0.21
Iodide	0.04–0.40	1.6	±0.07

The half-cell reaction of NCN can be written as

$$NCN + H^+ + e^- \rightleftharpoons NA + Cl^-$$

[NA: Nicotinamide].

The formal redox potential of [NCN]/[NA] couple has been found to be +1.02 V at 25°C. All the reductants undergo the same oxidation scheme as reported earlier[3].

The smoothness and appreciable change near the equivalence points in all the titration curves most probably suggest that NCN functions as an effective oxidiser for these reductants.

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