References

1. Korkisch, J.: Modern methods for the separation of rarer metal ions. Oxford: Pergamon Press 1969.
2. Sandell, E. B.: Colorimetric determination of traces of metals, 3rd ed. New York: Interscience 1959.

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Bromometric Determination of o-Ethoxybenzamide

Bromometrische Bestimmung von o-Äthoxybenzamid

Best. von o-Äthoxybenzamid; Bromometrie

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o-Ethoxybenzamide has its therapeutic value as an analgetic, antirheumatic and antipyretic agent. Usually this substance is evaluated through the determination of nitrogen content by Kjeldahl method. It has been found that o-ethoxybenzamide can be determined in a simple and rapid way by bromination. The quantity of bromine consumed suggests monosubstitution in the aromatic ring.

The reaction product is in accordance with formula I as could be shown by infra-red and proton-magnetic resonance spectrometry. The bromine substitution does not take place

in the 3rd position as well to give a disubstituted product. This is, for example, the case with salicylamide, where a dibromo-(3,5) substituted salicylamide is obtained on bromination. It could be only explained partly because of the immediate precipitation of the reaction product, and hence removal from the equilibrium state, and partly in steric terms. This is also in agreement with the results [1] where a 2,4-dibromoacetanilide, but not the 2,6-dibromoproduct is obtained. Even a 100% excess of bromine and a prolonged time of bromination (1 h) did not lead to a dibromo-substituted ethoxybenzamide.

The error of the method is $\pm 1\%$.

Procedure. Weigh 100–200 mg of o-ethoxybenzamide accurately in a stoppered Erlenmeyer flask, dissolve in 10 ml of methanol, add successively 50–75 ml of 0.1 N

KBrO₃ solution, 1 g powdered KBr and 15 ml of 6 N HCl and stopper the flask quickly. At this stage a precipitate is separated. Leave the flask in the dark and cold with occasional stirring for about 30 min, then add 10 ml of dichloromethane to dissolve the precipitate and 10 ml of 10% KI solution, stopper the flask and leave aside for 5 min. Titrate the iodine liberated with 0.1 N Na₂S₂O₃ solution using starch as indicator.

1 ml of 0.1 N KBrO₃ solution is equivalent to 8.26 mg of o-ethoxybenzamide.

Instead of methanol also glacial acetic acid [1] can be used for dissolving the sample, yet this causes slightly higher results.

Reference

1. Kertész, P., Förhécz, K.: Z. Anal. Chem. 258, 288 (1972).

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Titrimetric Determination of Indigo with Potassium Chlorate

Titrimetrische Bestimmung von Indigo mit Kaliumchlorat

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The applicability of potassium chlorate as a reagent for the direct oxidimetric titration of indigo has been studied.

Experimental

Indigo Sulphonate. About 1 g of pure indigo sample was sulphonated by digestion with 15 ml of conc. sulphuric acid at 90°C for 45 min. The reaction mixture was cooled, diluted to 500 ml with double distilled water and standardised according to the procedures of Allen [1] and Sastry and Gopala Rao [2].

Recommended Procedure. An aliquot of indigo sulphonate is taken in a 150 ml pyrex titration cell. Enough conc. hydrochloric acid and distilled water are added to give an overall acidity of 7–8 M when diluted to 50 ml. (This is necessary to ensure a sufficiently fast reaction.) The mixture is stirred well and titrated with 0.02 N potassium chlorate solution. The titration is carried out rapidly in the beginning and slowly towards the equivalence point allowing 15–20 sec after the each addition of a drop of the reagent. The end point is denoted by the sharp colour change from blue to pale yellow. The experimental values are to be multiplied by a factor of 1.078 to correspond to the oxidation of indigo sulphonate to isatin. The relative error of the titration is 0.3%.