Dethioacetalisation with Ceric Ammonium Nitrate

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Summary A ready dethioacetalisation procedure employing ceric ammonium nitrate in aqueous acetonitrile at room temperature is described.

The value of the thioacetal group for temporary masking the carbonyl function has increased recently through the development of versatile synthetic method based on the 1,3dithian carbanion.1 However, procedures for direct regeneration of carbonyl compounds from dithioacetals are still limited; methods most frequently used involve mercury(II) salts and N-halogenosuccinimides. Chloramine T has also been used,² and dethioacetalisation via S-alkylation has been reported.3

In a study of the oxidation of organic compounds by ceric ammonium nitrate (CAN) we have discovered a simple and convenient alternative. Thus, treatment of 1,3-dithiolans or 1,3-dithians (1.0 mmol) in 75% aqueous acetonitrile (12 ml) with CAN (4.0 mmol) at room temperature for 3 min, followed by dilution with water and extraction with ether, gave the parent carbonyl compounds in good yields (see Table). Pure products were isolated by distillation, or TABLE. CAN cleavage of dithioacetals

| 1,3-Dithiolans | Aldehyde or ketone Benzaldehyde ⁵ Acetophenone ⁶ Benzophenone ⁸ Cyclohexanone ⁶ | Yield (%) of parent compound 73.4^{a} 80.0 85.7 87.0^{a} |
|----------------|---|--|
| 1,3-Dithians | Methyl 3'-oxononyl ketone' Phenylacetone ¹ Cycloheptanone ¹ | 80·0 85·0 70·0 |

a Yield and identification based on isolated 2,4-DNP.

filtration through a neutral alumina column and recrystallisation, or as their 2,4-dinitrophenylhydrazones (2,4-DNP). This work was supported by the National Research Council of Canada.

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