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Physico-chemical Products and Oxidation of Some Organic Compounds with Chromium Oxidants

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Abstract

In the present work, the complexed oxidation products of propane-1, 2-diol and crotonaldehyde with TBC and TAC in various molar ratio have been studied. Out of the elements present (C, H, O & Cr) carbon and hydrogen were estimated instrumentally whereas chromium was determined by volumetric analysis. The possible formulation for the different products (complexes) obtained were tried with the help of empirical formula and the infrared spectra of the complexed products. We have taken TBC and TAC as oxidants for simple organic compounds like diols and enals like propane-1, 2-diol and crotonaldehyde. The products formed were analysed on the basis of elemental analysis, estimation, spectroscopic and thermal studies.

Keywords: Thermal, Chromium, Complex, Oxidation. Products

Chromium compounds exist in unusually large number of oxidation state (0 to VI). Chromium compounds like chromium oxides, chromates, dichromates etc., have been used since very long for the oxidation of both organic as well as inorganic compounds. Cr (Vi) containing oxidants are widely used in modern organic synthesis for the oxidation of a large number of compounds. Extensive work has led to the development of a number of such oxidants. e.g.:

1. Dipyridine chromium (VI) oxide[1]
2. Chromium trioxide-3,5-dimethyl pyrazole complex[2] (Collins Reagent)
3. Pyridinium chlorochromate (PCC)[3]
4. Pyridinium dichromate (PDC)[4]
5. 2,2′- bipyridinium chlorochromate (BIPCC)[5]
6. Pyridinium flourochromate[6,7] (PFC)
7. Quinolinium flourochromate[8]
8. Quinolinium chlorochromate 3, 5-dimethyl pyrazolium flourochromate
9. 2,6-dicarboxy pyridinium chlorochromate,[9,10]
10. N-methyl pyridinium chlorochromate[11]
11. Tetramethyl ammonium flourochromate (VI) (TMAFC)[12]
12. N-methyl benzyl ammonium flourochromate (VI) (MBAFC) [13]
13. Bi-tert-butyl chromate (TBC)
14. Bi-tert-amyl chromate (TAC) etc.

For the past century researchers have give a considerable attention to the field of oxidation. In modern organic synthesis, oxidation of an organic substrate the field of oxidation. In modern organic

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synthesis, oxidation of an organic substrate For the past century researchers have given a considerable attention to in anhydrous and aprotic solvent under mild and neutral conditions is important. Therefore synthetic organic chemists pay attention to the quest of non classical oxidants. In recent years, investigators have been looking for the oxidants with the characteristics like lower cost, higher yields, better selectivity, ability to perform in mild and neutral conditions, easy preparation, low toxicity and short reaction time etc. This has led to the preparation of variants of chromium (VI) as enumerated on page 1. As regards Mn (VII) based oxidants, most of them are so powerful, that controlled reactions are very difficult. Their solubility in organic solvent is the other big problem. Mn (VII) oxide, $Mn_2 0_7$, though reported in the literature, it is difficult to prepare as well as store.

RESULTS AND DISCUSSION

The suitability of Cr (VI) based oxidants, is underlined on the basis on the fact that they can be prepared easily. The controllability of the reaction is again a merit associated with Cr (VI) oxidants. However, literature survey reveals that not all of them have the same rating.

Keeping in view, the synthetic importance of Chromium (VI) based oxidants in the modern organic chemistry, the workers of late have widely studied their application and mode of action. To mention a few of the new Chromium VI based oxidants - di tertiary butyl Chromate. tertiary arnyl chromate, di-isopropyl Chromate, Chromium Peroxide etheate, pyridine-chromium peroxide, 2,2′bipyridyl chromium Peroxide etc. can be listed. These oxidants have been used to divide various classes of organic compounds and their merits and demerits have been studied.

As far as peroxo oxidants of Chromium (VI) are concerned, only 2,2 bipyridyl chromium peroxide and pyridine chromium peroxide have been reported to be successful as versatile oxidants for the organic substrate. However, their uses may be limited on the ground that:

1. They are not very much stable at room temperature.
2. The reactions may be accompanied with explosions.

H. Firouzabadi *et al.* have reported the non suitability of dipyridine chromium (VI) oxide and pyridine chlorochromate. The disadvantages associated with dipyridine chromium (VI) oxide are:

1. It must be used in large excess (molar ratio five or six).
2. It is unstable.
3. It is highly hygroscopic.
4. It is prepared by a dangerous procedure and can ignite spontaneously.
5. It shows poor selectivity in the oxidation of primary alcohols to aldehydes.

Similarly, a lot of difficulties have been reported in the use of pyridine chlorochromate as an oxidant like —

1. Its acidity is not suitable for the acid sensitive compounds and consequently buffering of the reaction mixture is essential.
2. It is hygroscopic.
3. In the oxidation of some classes of organic compounds, it has been found either ineffective or slow in reaction.

The prospect of diisopropyl chromate as an efficient and versatile oxidant for organic compounds is doubtful on the ground that the reaction takes a long time and it is very difficult to isolate and analyse the product.

Detailed studies of the previous works done by the chemists attracted them towards the use of ditertiary butyl chromate (TBC) as an oxidising agent whic has been found to be very useful in the oxidation and degradation of organic compounds. TBC has also been used in paper chromatography for the identification of gas phase passivation of gene to protect against corrosion. The merits associated with application of TBC as oxidant for organic compounds may be summarised below:

1. It is stable at room temperature.
2. Its preparation does not pose any problem.
3. The yield is satisfactory.
4. Reaction time for alcohol etc is very short.

5. The oxidation does not need higher ratio of the oxidant (in the present work the maximum ratio is substrate: oxidant 1 : 2).

However, in spite of detailed works touching various aspects of TBC, few important sides related to its character have not been given due importance i.e.

1. Oxidation of simple organic compounds by TBC.
2. The study of the oxidation products complexed with the reduced (or not reduced) chromium.
3. Thermal stability of the complexes and compound.
4. Magnetic behaviour of these complexes etc.

These aspects have been studied in Ranchi University by Dr. G. D. Mishra *et al.* But a lot more can be done in this regard.

In the present work, the complexed oxidation products of propane-1, 2-diol and crotonaldehyde with TBC and TAC in various molar ratio have been studied. Out of the elements present (C, H, O & Cr) carbon and hydrogen were estimated instrumentally whereas chromium was determined by volumetric analysis. The possible formulation for the different products (complexes) obtained were tried with the help of empirical formula and the infrared spectra of the complexed products. We have taken TBC and TAC as oxidants for simple organic compounds like diols and enals like propane-1, 2-diol and crotonaldehyde. The products formed were analysed on the basis of elemental analysis, estimation, spectroscopic and thermal studies.

CONCLUSION

The method adopted is quite suitable for the determination of the path of the reaction as by using the different ratios of the oxidant and studying the complexed products, it is quite possible to ascertain the mechanism because the complexation of the product with chromium in different ratios implies the arrest of oxidation products at different stages of oxidation. In this way much light can be thrown on the path of the reaction which is main aim of the present work.

Moreover, the effect of solvent on the rate of reaction and the nature of product may also be an interesting aspect. The important solvents taken up for the purpose are THF, Dioxane, dichloromethane & acetonitrile. DMSO was also tried as solvent but it was not found to lead any fruitful result.

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