INTERNATIONAL JOURNAL OF ADVANCE RESEARCH, IDEAS AND INNOVATIONS IN TECHNOLOGY

ISSN: 2454-132X
Impact factor: 4.295
(Volume3, Issue4)
Available online at www.ijariit.com

A Study on Direct Oxidation of O-Toluidine by Potassium Bromate

Tapas Ghosh
Research Scholar, Department of Chemistry
Swami Vivekanand University, Sagar, M.P., India
tapasdps67@gmail.com

Abstract*: O-Toluidine is a very specific reagent used widely in various Laboratory. It finds a place in the forensic medicines laboratory where human blood stains on various objects are tested and used for crime detection. Besides these O-Toluidine is utilized in the synthesis of many useful organic compounds. A valuable report has been published on the germicidal action of O-Toluidine. Thus selection of O-Toluidine for mechanistic studies seems interesting and inviting as well.*

Keywords: Potassium Bromate, O-Toluidine.

EXPERIMENTAL: All the chemicals used were AR Grade; O-Toluidine E. Merck, Potassium Bromide, Sulphuric Acid A.R

O-Toluidine is a mild reducing agent.It would react with Potassium bromated rather favourably but not exactly in quick succession. A moderate concentrated of Sulphuric Acid at an elevated temperature is an appropriate choice for causing oxidation of O-Toluidine effectively. Potassium bromated in Sulphuric Acid
Medium liberates bromated ion (BrO_3^-) which may exist monoprotonated acid ($HBrO_3$).

$$KBrO_3 + H_2SO_4 \rightleftharpoons KHSO_4 + HBrO_3$$

$$HBrO_3 \rightleftharpoons H^+ + BrO3^-$$

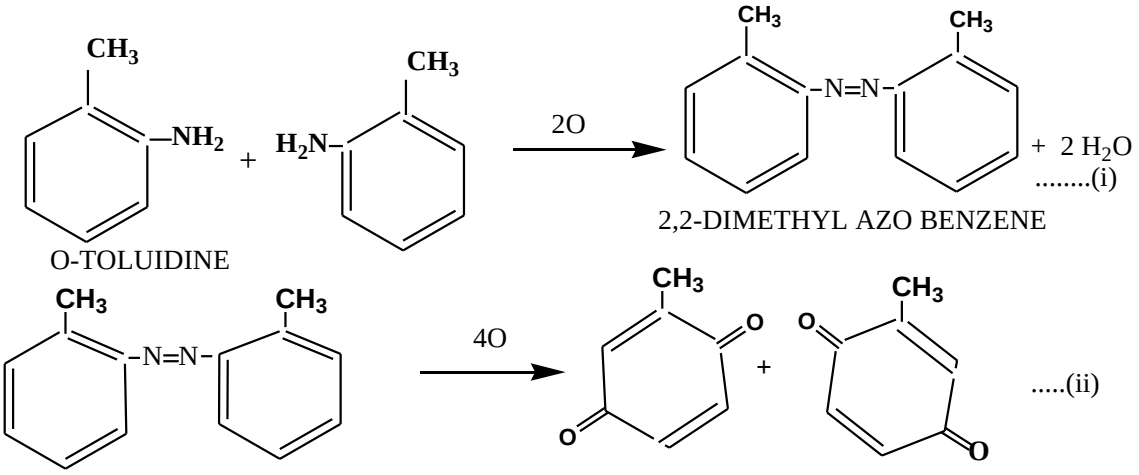
In our case, Sulphuric Acid (1M) has been used so the question of BrO_2^+ as oxidizing species may not arise.
0.01 mole (1.675 gm.) of $KBrO_3$ was taken in a clean and dry conical flask (250 ml). 0.01 mole of O-Toluidine (1.070 gm.) was added carefully over the oxidant.
The adhering substance was washed with H_2SO_4 (1M). The washing was transferred to the reaction vessel containing the reactants. Later on, 100 ml of H_2SO_4 (1M) was poured into it. The reactants were mixed at room temperatures (19° C). A dark brown reaction mixture containing some solid substance was obtained. Reddish brown vapours of Bromine gas was available in the flask. Again 10 ml. H_2SO_4 (1M) was added to dissolve the residue. It was shaken vigorously for fifteen minutes. A reddish brown reaction mixture in solution was obtained.

Now, the reaction mixture was immersed in a boiling water bath maintained at (80-90°c) for three hours continuously. It was then removed from water bath and allowed to stand at room temperature (18-19° C) for five days. The reaction mixture became dark red. A dark brownish solid with a radish layer at the top was obtained. This product was filtered and the product was dried within the folds of filter paper. The purification of this final product was done by steam distillation, thereupon (2-Methyl-1, 4- Benzoquinone M.F $C_7H_6O_2$, M.W.122) was obtained along with water being sparingly soluble in cold water. It was filtered and pressed and recrystallised from aqueous – alcoholic medium. This gave yellowish fine needles. It was left for two days in a desiccator and then subjected to m.p. determination by direct thermal analysis (sample-DBr1.).
This corresponded to m.p. 67°c against the theoretical m.p. 69°c (curve c).

The final confirmation was sought by infrared spectroscopy (curve No-IR1). Infrared spectrophotometer which was utilized for this purpose was Perkin-Elmer. Model – 598 (U.K.). D.T.A. was done in collaboration with Dr. R. P. Singh at central mining research station, Dhanbad by the courtesy of chemical section. Infrared analysis was carried out in the chemistry department of Banaras Hindu University, Varanasi, India.

During the direct oxidation experiment with an acidic potassium Bromate o-Toluidine seems to have been oxidized to 2- methyl 1,4 Benzoquinone via 2,2- Dimethyl Azobenzene.

The steps of the reaction are represented in (i) and (ii):



O-TOLUIDINE

2,2-DIMETHYL AZO BENZENE

2,2-DIMETHYL AZO BENZENE

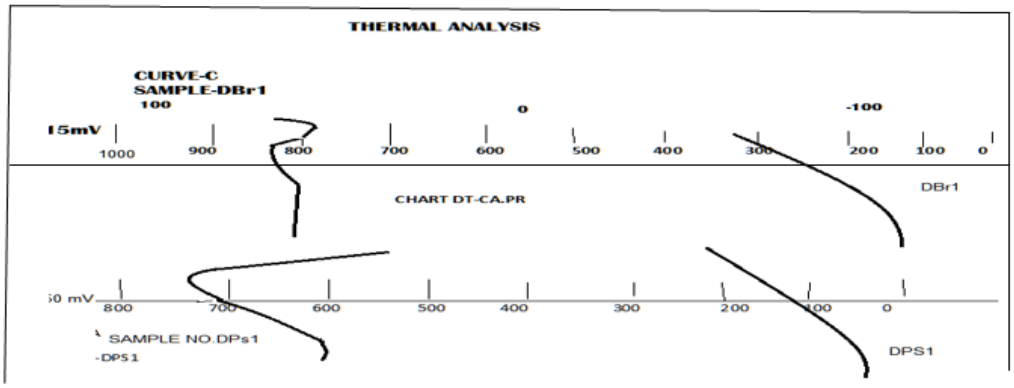
2-METHYL -1,4-BENZO QUINONE

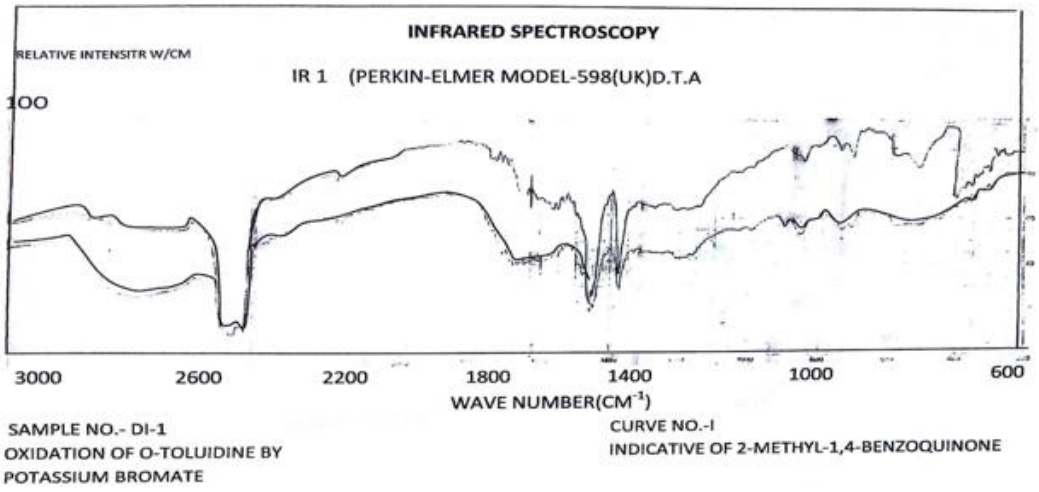
More clearly, the reaction (ii) may be viewed as:-

$$C_{14}H_{14}N_2 + 2KBrO_3 + 8H^+ \longrightarrow 2C_7H_6O_2 + 2KBr + 2H_2O + 2NH_3 \quad .....(iii)$$

2,2-Di Methyl Azo Benzene

2-Methyl-1,4-Benzo Quinone





SAMPLE NO.- DI-1
OXIDATION OF O-TOLUIDINE BY POTASSIUM BROMATE

CURVE NO.-I
INDICATIVE OF 2-METHYL-1,4-BENZOQUINONE

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