

DETERMINATION OF THIOUREA AND ITS DERIVATIVES WITH BROMINE MONO-CHLORIDE REAGENT BY MICRO-METHOD

*D.P. Prajapati¹ Ranveer Singh²

1. Department of Chemistry, Govt. P.G. College Seoni (M.P.)

2. Department of Chemistry. Satish Chandra P.G. College Baliya (U.P.)

Abstract- A survey of literature reveals that bromine mono-chloride has not been used for the determination of these compounds on micro scale. More a method has been developed for the determination of thio-urea with the use of bromine mono-chloride (BrCl) reagent.

KEYWORDS: Thio-urea, Bromine monochloride, Micro scale metho.

I. INTRODUCTION-

Thio-urea are generally used as preservatives, insecticides, rodenticides and in the pharmaceutical preparation. Many Thiourea derivatives possess antibacterial and antipyretics properties¹ as well. They are of great value in the characterization of Organic Compounds and are used in dyes, photographic film, plastics and textile industry and for the manufacture of chemical deposited radiation – detector materials. In these operation it is often necessary know the purity of particular thioureas derivatives sample and therefore, a suitable method for their easy would of great value. A number of methods have been proposed for the determination of Thioureas. Yadav and Jain¹⁶ performed coulometric estimation of Thiourea. Joshi¹⁸ demonstrated that tetravalent selenium can be used as quantitative oxidant for Thiourea. Erik Wernersson, Björn Stenqvist, Mikael Lund², The mechanism of cellulose solubilization by urea studied by molecular simulation, Y. Liu, W. Jian, J.Y. Wang, S. Hofmann, K. Shimizu³ structure of thiourea on copper. Fatma Karipcin, Murat Atis, Bahtiyar Sariboga, Asan Celik, Murat Tas⁴. Structural, spectral, optical and antimicrobial properties of synthesized 1-benzoyl-3-furan-2-ylmethyl-thiourea. Zerong Daniel Wang, Motoko Yoshida, Ben George.⁵

Theoretical study on the thermal decomposition of thiourea.

PRESENT WORK

A survey of literature reveals that bromine mono-chloride has not been used for the determination of these compounds on micro scale. More a method has been developed for the determination of thiourea with the use of bromine mono-chloride (BrCl) reagent. Most of the methods described above need sophisticated instruments and drastic reaction conditions; therefore, it was thought to develop a simple, quick and convenient method for the determination of the some Thio-urea and its derivatives.

Using Thiourea as the test sample, the effect of temperature was observed that the best recovery of the sample was obtained at a temperature of boiled water bath give best result of 40 °C (Table-I). 40 °C temperature for their complete oxidation and Reaction time 15 Minutes and the concentration was varied from 0.10N to 0.40 N and the Recovery of the sample was calculated. It was found that the best recovery was obtained by using 0.3 N concentration of the reagent

Micro determination of thio-urea and its few derivatives with 0.3N BrCl reagent. With the recommended procedure micro determination of thiourea Phenylthiourea α -Naphthyl thiourea, Allyl thiourea, o-ditolyl thiourea, Trio semi carbazide was carried out Table -1

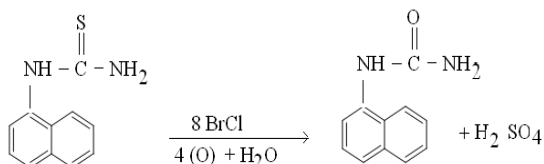
RESULT AND DISCUSSION:

As is evident from the table 11 the recommended procedure has been successfully applied for micro determination of Thiourea, recovery of the Sample is

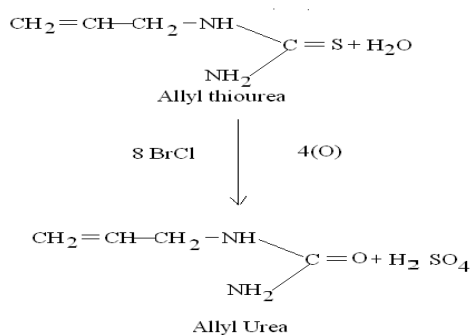
accurate up to 10 mg. of the sample Size but for the general procedure 1-5 mg. sample rise is sufficient. Thiourea was taken as the representative compound to study the effect of various variables on the reaction.

POSSIBLE COURSE OF REACTION

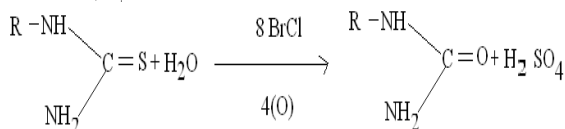
While studying the reaction time, It was observed that Thiourea derivatives containing electron withdrawing group like Naphthyl, Phenyl etc. are readily oxidized due to weakening of C=S bond by these groups thus Naphthyl Thiourea is oxidized in the following manner like phenyl Thiourea.



Similarly, Allyl thiourea reacts with eight equivalent of Bromine mono chloride giving rise to corresponding urea and Sulphuric acid.

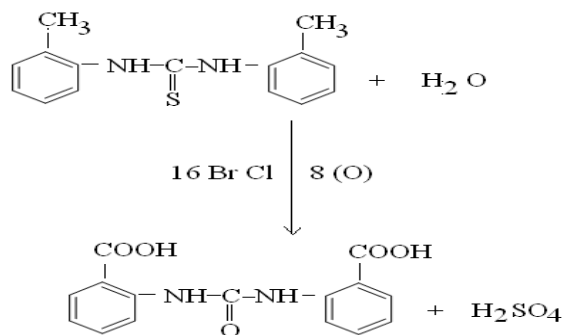


Based on above observation of a general reaction for the oxidation of all these Thiourea compound may be written in the following form.



Where R = H, C₆H₅, CH₂=CH-CH₂- Groups

As indicated in stoichiometry table o- ditolyl Thiourea consume 16 equivalent of bromine Mono chloride (BrCl) it may passing is oxidised in the following manner.



It was observe that the process of Urea and it derivatives due inform in the determination,

GENREL PROCEDUER

Aliquots containing 1-5 mg of the sample were taken in a 100-ml. Erlenmeyer flask followed by the addition of 6 ml of 0.3 N Bromine mono chloride reagents and 5 ml. of 10% Sulphuric acid. The reaction contents were shaken gently and kept on a boiling water bath for 20 minutes. After the reaction was over, the reaction mixture was cooled to room temperature. The unconsumed bromine mono chloride reagent was titrated against 0.025 N ferrous ammonium sulphate using N-Naphthyl anthranilic acid as an indicators. A blank experiment was also run under identical condition using all the reagent except the sample. Discovery of the sample was calculated by following expression.

$$\text{Mg. of the Sample} = \frac{M \times N (B - S)}{n}$$

Where -M= Molecular weight of the sample
 N= Morality of Ferrous Ammonium Sulphate
 B = Volume of Ferrous Ammonium Sulphate consume to titrate the blank Experiment.

S = Volume of Ferrous Ammonium Sulphate Consume to titrate the sample Experiment.

N = Number of mole of Bromine mono chloride reagent consumed per mole of the sample.

TABLE - 1: Micro determination of thiourea and its some derivatives with recommended procedure with 0.3N Bromine Mono Chloride reagent

Aliquots taken (ml.)	Amount present(mg.)	Reaction time (.Min)	Amount Recover(mg.)	Morality	Error %
Thiourea	1.0000	15	0.9975	8	-0.25
	3.0000		3.0066		+0.22
	5.0000		5.0120		+0.24
Phenyl Thiourea	1.0000	15	1.0042	8	+0.42
	3.0000		2.0046		-0.10
	5.0000		2.9900		-0.20
ó-Naphthyl thiourea	1.0000	20	1.0044	8	+0.44
	3.0000		2.9865		-0.45
	5.0000		4.9900		-0.20
Thiosem carbazide	1.0000	20	1.0120	12	+0.12
	3.0000		3.0120		+0.40
	5.0000		5.0335		+0.67
Allyl thiourea	1.0000	30	1.0042	8	+0.42
	3.0000		2.9865		-0.45
	5.0000		5.0120		+0.24
o-di – tolyl thiourea	1.0000	30	0.9975	16	-0.25
	3.0000		3.0120		+0.40
	5.0000		4.9900		-0.20

REFERENCE-

- Vincenzo Barone, Malgorzata Biczysko, Julien Bloino, Paola Cimino, Emanuele Penocchio and Cristina Puzzarini *Journal of Chemical Theory and Computation* **2015** Article ASAP.
- Erik Wernersson, Björn Stenqvist, Mikael Lund *Cellulose* **2015** 22 (), 991-1001
- Y. Liu, W. Jian, J.Y. Wang, S. Hofmann, K. Shimizu *Applied Surface Science* **2015** 331 (), 140-149.
- Fatma Karipcin, Murat Atis, Bahtiyar Sariboga, Hasan Celik, Murat Tas *Journal of Molecular Structure* **2013** 1048, 69-77.
- Zerong Daniel Wang, Motoko Yoshida, Ben George *Computational and Theoretical Chemistry* **2013** 1017 (), 91-98
- Abdel-Wahab BF, Awad GE, Badria FA (2011).
- Inorg. Chim. Acta* 359(12):3976-3984. Gacche RN, Jadhav SG (2012).
- Crys. Growth Des.* 4:1105-1107. Nikhili B, Shikha B, Anil P, Prakash NB (2012).
- Int. Res. J. Ph.* 3(7):24-28 Paramjeet KM, Dipak S, Arti D (2012).
- Int. J. Antimicrob. Age* 33(5):421-426. Venugopala KN, Rashmi V, Odhav B (2013).
- Res. Int. Article ID* 963248. Završnik D, Halolovic SŠ, Softic D (2011).
- R.Shakru, N j p Subhashini, S. Kumar, K. Shikvraj *J Chem. Pharm.Res.* 2010.2(1)38-46
- A.P.Mishra, R.K. Jain *J Chem. Pharm. Res.* 2010.2(6)51-61
- J.H.Deshmukh, M.N.Deshpande *J Chem. Pharm. Res.* 2011.3(3)206-212
- N S Dixit and C C Patel, *J Indian Chem. Soc.* 1997,54,176-180.

16. Yadavs K. L. And Jains S. K, The Proceeding of the National Academy of Science , See A , 34, 521, (1966)
17. Surendra , S. And K. I.,Yadavs,D. Phil. Thesis,Allahabad University , Allahabad.
18. Joshi. M.K. Anal, Chem. Acta., 14,509, (1956).
19. Tiwari R. D. and Pandey, Analyst , 94, 813, (1969) U.G.
20. Joshi M. K. Chem. Lity, 50, 1920, (1981).
21. Volhard J. Berichte , 7, 100 (1974).
22. Malaysian Journal of Analytical Sciences, vol. 15, no. 1, pp. 37–45, 2011.
23. Journal of Organic Chemistry, vol. 8, pp. 1443–1451, 2012.