Thermo. Titr. Application Note No. H-095

Title:	**Determination of Urea by Non-Aqueous Titration**

***Scope*:**	Determination of urea by titration with trifluoromethanesulfonic acid. The titration is suitable for full automation using an 814 Sample Processor.

***Principle*:**	Dissolution of urea in glacial acetic acid, and titration with standard 0.1mol/L trifluoromethanesulfonic acid in acetic acid using isobutyl vinyl ether as a thermometric endpoint indicator. (***Ref.***: E. J. Greenhow and L. E. Spencer (1973) *Ionic polmerisation as a means of endpoint indication in non-aqueous thermometric titrimetry. Part 1. The determination of organic bases.* Analyst, **98**, 81-89)

Reagents:	*Titrant:* 0.1mol/L trifluoromethanesulfonic acid in glacial acetic acid – Riedel de Haën (Sigma-Aldrich) cat. no.35317 *Endpoint indicator:* Isobutyl vinyl ether Aldrich cat. no. 278351 *Solvent:* Glacial acetic acid , A.R. *Test substance:* Urea (old reagent, opened bottle)

Method:	Basic Experimental Parameters:	
	Titrant delivery rate (mL/min.)	4
	No. of exothermic endpoints	1
	Data smoothing factor (DSF)	70
	Stirring speed (802 stirrer)	12
	A test solution of ~0.1mol/L was prepared by accurately weighing ~3g urea and dissolving with gentle warming in glacial acetic acid. The slution was cooled, and transferred quantitatively to a dry 250mL volumetric flask, and made to volume with glacial acetic acid. For demonstration purposes and highest precision, aliquots between 1 – 5mL were dispensed with a 10mL Dosino burette into a titration vessel together with 35mL glacial acetic acid from another Dosino. A 1mL dose of isobutyl vinyl ether was added to the vessel prior to the commencement of the titration.	

	For routine analysis, approximately 1g of urea could be weighed accurately directly into a 200mL volumetric flask, and gently warmed with 20mL glacial acetic acid. The flask would be allowed to cool, and then made to volume with glacial acetic acid. Aliquots of 5mL would be pipetted, and 1mL isobutyl vinyl ether added (this is equivalent to ~0.025g urea). 35mL glacial acetic acid would be added by Dosino prior to the commencement of the titration. Duplicate determinations may be made for highest accuracy.

Example:	***Purity of old urea reagent***
	95.46±0.12% (n=8)

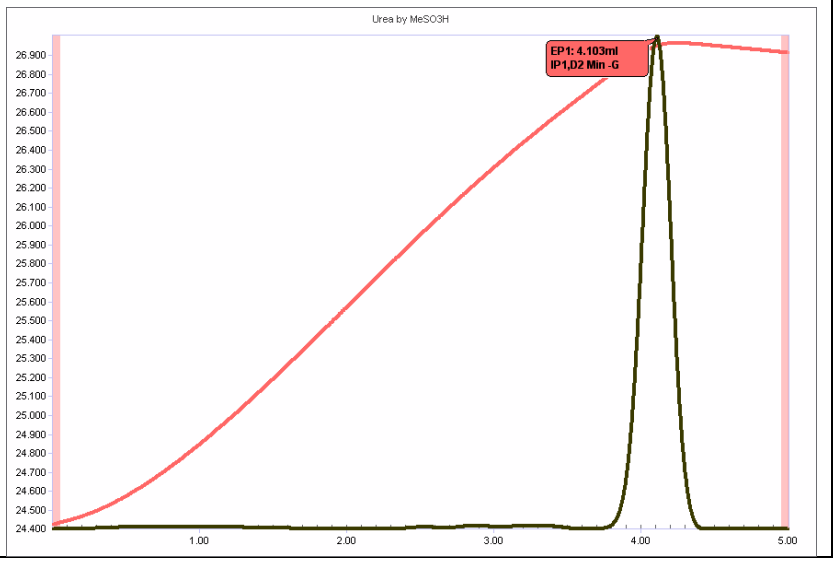
Calculations:

$$(NH_2)_2CO\% = \frac{((\text{Titre, mL} - \text{blank, mL}) \times F_3CSO_3H \text{ mol/L} \times 60.055 \times 100)}{(\text{aliquot, g} \times 1000)}$$

(urea is monobasic in this titration)

Thermometric Titration Plot:

Legend:
Red = solution temperature curve
Black =second derivative curve (for endpoints)



Determination of blank:

Titration blank =

y-intercept = 0.2444mL