Estimation of Limit of Detection and Limit of Quantitation in Titrations for Weak Dissociable Cyanide in Water and Free Cyanide

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APPLICATION Note 2

Abstract

No concentration should ever be reported as "0". The limit of detection of free cyanide by this titration method should be determined and if the value obtained on a sample is less than the LOD, the result should be reported as "not detected" or <LOD. It is an important analysis to perform as cyanide is used for extraction of gold in mining and the extent that this anion may be reporting to environment needs to be known.

1. Weak Dissociable Cyanide in Water (WAD)

An important analysis required in the gold mining industry where cyanide is used to extract gold is that of WAD free cyanide. Metal cyanide complexes, *e.g.* Zn, Cd, Cu, Ni and Ag, are stable; however at a pH of 4.5 these complexes release free cyanide into the environment. Free cyanide is toxic to human and animals.

1.1 Method of Estimation of WAD

A 50ml sample solution is taken using a pipette and transferred into a 500 ml Erlenmeyer flask. Approximately 0.1g of lead carbonate is added to the sample and swirled to dissolve; this step removes sulphide from the sample solution. The sample is then filtered and washed with distilled water into a second Erlenmeyer flask. Using a dispenser 10ml ammonia solution and approximately 3g of zinc powder followed by 10ml of 5 mole/dm^3 of sodium hydroxide solution are added. The flask is then stoppered and stirred using a magnetic stirrer for 15 minutes. Following this solution is filtered into another clean

Erlenmeyer flask. A few drops of potassium iodide indicator solution at 10% (v/v) concentration are added to the sample solution. The titration is carried out against a black background with 0.02 mole/dm^3 standard silver nitrate solution to a white cloudy end-point. The volume of the silver nitrate titrated is used to calculate the free cyanide concentration.

2. Free Cyanide in Water

This method is used for the determination of measuring free, *i.e.* non-complexed cyanide and hydrocyanic acid in drinking water, natural surface waters, domestic and industrial wastewaters, and in soil extracts. It is an important analysis to perform as cyanide is used on the plant and the extent of this anion that may be reporting to environment needs to be known.

2.1 Method of Estimation of Free Cyanide in Water

Before extracting a sample for analysis the sample bottle needs to well mixed. A blank solution was prepared by pipetting 50ml of distilled water and transfer into an Erlenmeyer flask. A 50ml of sample solution was taken using a pipette and transferred into a second Erlenmeyer flask. A few drops of rhodanine indicator is added to both to the sample solution and blank. The sample was titrated with silver nitrate to a colour change end-point of pink to pale yellow.

2.2 Estimation of the Limit of Detection and Limit of Quantitation

The limit of detection (LOD) and limit of quantitation (LOQ) of free cyanide is determined by titrating at least 8 to 10 replicate blanks (50ml of distilled water). From these data, the average of the replicates is found and the standard deviation calculated (Table 1). The LOD is found as:

$$LOD = 3 \; x \; standard \; deviation \; of \; n \; blank \; measurements \; in \; mg/l$$

And, the LOQ is found as:

$$LOD = 10 \; x \; standard \; deviation \; of \; n \; blank \; measurements \; in \; mg/l$$

The LOD is therefore:

$$LOD = 3 \; x \; 0.191 \; mg/l = 0.6 mg/l$$

And, LOQ is:

$$LOD = 10 \; x \; 0.191 \; mg/l = 2 mg/l$$

Therefore, the LOD= 0.6mg/l   and the LOQ=2mg/l. Results therefore less than 0.6mg/l should be reported as "not detected", and results less than the LOQ report as "detected" and results $\geq$LOQ, report the results e.g. 10.4mg/l.

Table 1. Replicates of a 9 blank titrations of distilled water for the estimation of the limit of detection of free cyanide in water.

	NaCN (mg/l)
	2.43
	2.49
	2.58
	2.45
	2.52
	2.35
	2.01
	2.69
	2.35
Average=	**2.43**
s=	**0.191**

3. How to cite this application note

Cite this application note as:

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