Short Communication

Determination of isocyanuric acid, ammelide, ammeline and melamine in crude isocyanuric acid by ion chromatography

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ABSTRACT

A reliable method for the determination of isocyanuric acid, ammelide, ammeline and melamine in crude isocyanuric acid is presented. The method involves using an ion chromatograph with an Omnipac PCX 500 column, 100 mM potassium chloride–200 mM hydrochloric acid–5% acetonitile solution as mobile phase and a UV detector at 215 nm.

INTRODUCTION

Isocyanuric acid is used mainly as a swimming pool stabilizer and as an intermediate in the preparation of its chloro derivatives. A reliable analytical method was required to determine the isocyanuric acid content and the impurities, ammelide, ammeline and melamine in crude isocyanuric acid. The structures of these compounds are shown opposite.

There are only a few reports relating to this problem. Although the thin-layer chromatography method [1,2] is satisfactory for qualitative analyses, the method is not entirely suitable for quantitative purposes. A gas chromatography method [3] required a preliminary derivatization procedure, which is considered a disadvantage because of the longer time required for sample

Melamine   Ammeline

Ammelide   Isocyanuric Acid
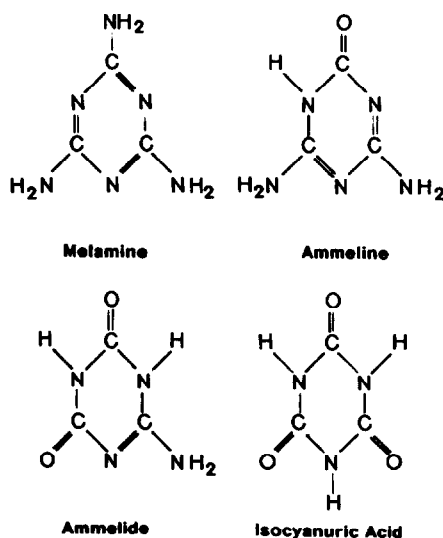
preparation and the concomitant source of analytical errors.

Two HPLC methods [4,5] were also found to

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be unsuitable for this particular application. The first one [4] (using a Lichrosorb RP-18 column, potassium phosphate buffer–methanol gradient, and a temperature of 2°C) produced broad, tailing peaks that were not completely separated. Although the second one [5] (using a Zorbax NH_2 column and acetonitrile–phosphate buffer solution) gave improved separations, the chromatograms were difficult to reproduce. This was possibly due to batch differences in Zorbax NH_2. Similar problems were also experienced with a method developed on a Diol column, which failed after replacing the column with one from a different batch. This implies that the above methods are very sensitive to small changes in stationary phase and are therefore unsuitable for routine use.

The method presented here is based on strong interaction between amines and cation-exchanger stationary phase and is considered to be an improvement upon existing procedures.

EXPERIMENTAL

Instrumentation

A Dionex 4500i ion chromatograph system equipped with a Dionex variable-wavelength detector VDM-2 at 215 nm, a Spectrophysics 4270 integrator, a manual-pneumatic injector with 10-μl sample loop, a Dionex Omnipac PCX-500 guard column (50 × 4.0 mm I.D.) and a Dionex Omnipac PCX-500 analytical column (250 × 4.0 I.D.) was used.

Chemicals and reagents

The isocyanuric acid, ammelide, ammeline and melamine were analytical-grade quality.

The mobile phase components, hydrochloric acid and potassium chloride, were analytical reagent grade, acetonitrile was HPLC grade and the water was purified through a Milli-R04/Milli-Q plus Millipore water purification system. The mobile phase was prepared to contain 100 mM potassium chloride, 200 mM hydrochloric acid and 5% acetonitrile in deionized water.

Procedure

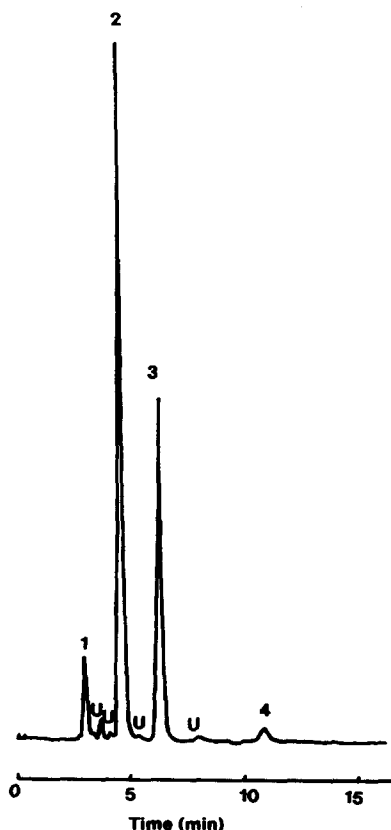Samples were dissolved in the mobile phase and filtered through a 0.45-μm nylon filter prior

Fig. 1. Ion chromatogram of crude isocyanuric acid. Peaks: 1 = Isocyanuric acid; 2 = ammelide; 3 = amr.ieline, 4 = melamine; U = unknowns. Conditions: column, Omnipac PCX-500; injection, 10 μl; mobile phase, 100 mM potassium chloride–200 mM hydrochloric acid–5% acetonitrile. Flow-rate, 1.0 ml/min; UV detection at 215 nm.

to injections. The conditions for the determination are shown in Fig. 1.

The calibration graphs for isocyanuric acid, ammelide, ammeline and melamine were found to be linear within the following analytical ranges: 50–500 mg/l, 10–200 mg/l, 2–70 mg/l and 0.3–10 mg/l, respectively. The correlation coefficients were 0.99996, 0.99993, 0.99996 and 0.99998, respectively.

RESULTS AND DISCUSSIONS

The accuracy of the method was tested by adding known concentrations of isocyanuric acid, ammelide, ammeline and melamine to a typical crude isocyanuric sample dissolved in eluent and

TABLE I

RESULTS OF ISOCYANURIC ACID, AMMELIDE, AMMELINE AND MELAMINE IN A CRUDE ISO-CYANURIC ACID SAMPLE

Compound	Concentration[a] (%, w/w)	R.S.D. (%)
Isocyanuric acid	70.4	2.6
Ammelide	23.9	2.1
Ammeline	4.2	1.2
Melamine	0.1	5.0
Total	98.6	

[a] Mean of five determinations.

determining the total amounts present. The recoveries varied between 100 and 102%, which indicated that the accuracy of this method was satisfactory.

The isocyanuric acid, ammelide, ammeline and melamine content of a typical crude isocyanuric acid sample is presented in Table I. A typical chromatogram of this sample is shown in Fig. 1.

As shown in Table I the total content is 98.6% (w/w). The balance of 1.4% can be related to the unidentified impurities clearly visible in Fig. 1.

It was also found that trichloroisocyanuric acid (TCICA) and dichloroisocyanuric acid sodium salt (DCICA) elute at the same time as isocyanuric acid. This can be explained by the conversion of all isocyanuric acid chloro derivatives into isocyanuric acid itself under existing chromatographic conditions ($100 \ mM$ hydrochloric acid) [6]. The detection limits for isocyanuric acid, ammelide, ammeline and melamine were 50, 1.5, 0.6 and 0.3 ng, respectively.

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