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Visual detection and sequential injection determination of aluminium using a cinnamoyl derivative



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ABSTRACT

A cinnamoyl derivative, 3-[4-(dimethylamino)cinnamoyl]-4-hydroxy-6-methyl-3,4-2H-pyran-2-one, was used as a ligand for the determination of aluminium. Upon the addition of an acetonitrile solution of the ligand to an aqueous solution containing Al(III) and a buffer solution at pH 8, a marked change in colour from yellow to orange is observed. The colour intensity is proportional to the concentration of Al(III); thus, the 'naked-eye' detection of aluminium is possible. The reaction is also applied for sequential injection determination of aluminium. Beer's law is obeyed in the range from 0.055 to 0.66 mg L⁻¹ of Al(III). The limit of detection, calculated as three times the standard deviation of the blank test ($n=10$), was found to be 4 µg L⁻¹ for Al(III). The method was applied for the determination of aluminium in spiked water samples and pharmaceutical preparations.

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1. Introduction

Aluminium is a widespread metal occurring naturally in bauxite rock, silicates and cryolite. Its path into drinking water and food occurs through a transmission chain in nature caused primarily by human activity [1]. Aluminium and its compounds have extensive applications in a variety of fields, mainly in the manufacture of alloys, glass and ceramics and in the automobile and transport industries. It is also used in cosmetics production and the pharmaceutical and food industries [1,2]. Aluminium salts are used as coagulants for the treatment of drinking water. According to WHO, the tolerable value of aluminium in drinking water is limited to 0.2 mg L⁻¹ [3]. The continuous increase in human activity leads to soil acidification and thus contributes to higher levels of mobile forms of aluminium. Hartwell and Pember [4], in

1918 studied the toxic effect of aluminium on plants due to increasing acidity in the soil substrate, and thus far several studies have been published on this topic [5–7]. The effect of aluminium on the human body and human health as well as aluminium intake through cosmetic preparations [8] and pharmaceutical preparations [9–11], particularly in childhood vaccines [12–14], is still being discussed. Aluminium has a potential neurotoxic effect [15], and its intake by the human organism is connected with, for example, Alzheimer's disease [16–19], autism [20] and breast cancer [8]. Therefore, the determination of aluminium in environmental, pharmaceutical and cosmetic preparations is very important and greatly needed [21].

Several articles concerning the determination of aluminium using flow analysis and atomic spectrometry [22], fluorimetry [23,24], or spectrophotometry [25–27] have been published. Developing visual detection procedures can be considered as one of the most interesting topics in analytical chemistry today. Several articles on the visual detection of a variety of cations and anions [28–33], including aluminium, have been published (Table 1).

In this work, the reaction of Al(III) with 3-[4-(dimethylamino)cinnamoyl]-4-hydroxy-6-methyl-2H-pyran-2-one ligand (L) was employed (Fig. 1) for the visual detection and sequential injection determination of aluminium. The ligand is a cinnamoyl derivative belonging to the family of cinnamoyl pyrones. Cinnamoyl pyrones are known as medicaments and as intermediate products of medicament synthesis [34]. The structure and spectral properties of cinnamoyl pyrones were discussed by Tykhanov et al. [34,35].

Abbreviations: L, 3-[4-(dimethylamino)cinnamoyl]-4-hydroxy-6-methyl-2H-pyran-2-one (ligand); SV, Selection Valve; HC, Holding Coil; RC, Reaction Chamber; SP, Syringe Pump; B, Buffer solution; A, Acetone; AN, Acetonitrile; SVo, Sample Volume; LR, Linear Range; BR, Bromopyrogallol Red; n-TCTMAB, n-Tetradecyltrimethylammonium bromide; CAS, Chromeazurol S; CPC, Cetylpyridiniumchloride; ARS, Alizarin Red S; PV, Pyrocatechol Violet; MTB, Methyl Thymol Blue; A DBM, Arsenazo DBM; ECR, Eriochrome cyanine R; CTMAB, Cetyltrimethylammonium bromide; AQ, Anthraquinones.

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Table 1
Comparison of the developed visual method of Al(III) with those previously published in the literature.

Ligand	Comments	Sample	CC	LOD	Ref.
3-[4-(Dimethylamino)cinnamoyl]-4-hydroxy-6-methyl-2H-pyran-2-one	Water:AN (4.3:0.7, v/v); pH 8.0; SIA UV-vis detection (CC: up to 0.66 mg L ⁻¹ , LOD: 4 µg L ⁻¹)	Tap and spring water	0.11–0.88 mg L ⁻¹	0.11 mg L ⁻¹	This work
5-[(2-Hydroxy-5-nitro-benzylidene)-amino]-1H-pyrimidine-2,4-dione	DMSO:water (95:5, v/v)	–	–	25–50 µmol L ⁻¹	[36]
2-(2-(2-Hydroxyethoxy)ethoxy)ethyl 8-propoxyquinoline-2-carboxylate	AN:water (95:5, v/v)	Tap water	0–23 µmol L ⁻¹	0.8 µmol L ⁻¹	[37]
5-[(2-Hydroxynaphthalen-1-yl)methylene]amino]pyrimidine-2,4(1H,3H)-dione	UV-vis detection (CC: up to 50 µmol L ⁻¹ in AN) Fluorescent detection (CC: 0–2 equiv Al ³⁺ ; LOD: 3.2 × 10 ⁻⁷ mol L ⁻¹ in AN, 1 × 10 ⁻⁶ mol L ⁻¹ in water)	–	–	–	[38]
Under UV-light					
2,2'-Dihydroxyazobenzene	Spot test on hydrophobic filter paper; pH 6.5	–	–	2 × 10 ⁻⁸ mol L ⁻¹	[39]
2,2'-Dihydroxyazobenzene	The hydrophobic surface of the octadecylsilanized silica thin layer	Tap and river water, tea	–	1 µg L ⁻¹	[40]
4-(8'-Hydroxyquinolin-5'-yl)methyleneimino-1-phenyl-2,3-dimethyl-5-pyazole	10% methanol solution; pH 4.5; Fluorescent detection (CC: 0–22 µmol L ⁻¹ ; LOD: under 10 ⁻⁷ mol L ⁻¹)	–	–	–	[41]
2-Hydroxynaphthylidene-(8'-aminoquinoline)	DMF solution; Fluorescent detection (CC: 0–16 equiv Al ³⁺ ; LOD: 1 µmol L ⁻¹)	–	–	–	[42]
Methyl pyrazinylketone benzoyl hydrazone	Ethanol solution; Fluorescent detection (CC: 0–1 equiv Al ³⁺ ; LOD: 10 ⁻⁷ mol L ⁻¹)	–	–	–	[43]

CC, Calibration curve; AN, Acetonitrile; –, data not found in paper.

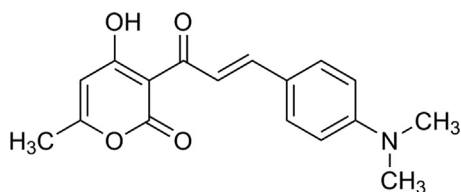


Fig. 1. Structure of 3-[4-(dimethylamino)cinnamoyl]-4-hydroxy-6-methyl-2H-pyran-2-one.

To the best of our knowledge, there have been no publications on the application of this ligand to the spectrophotometric determination of analytes.

2. Experimental

2.1. Reagents

All reagents and solvents were of analytical grade purity. Distilled water was used throughout the experiment. A stock aqueous solution containing 10 mmol L⁻¹ of Al(III) was prepared by dissolving 0.1876 g of Al(NO₃)₃ · 9H₂O (Centralchem, Slovakia) in water and diluting to 50 mL. The working solutions of various concentrations of Al(III) were prepared by step-wise dilution of the stock solution. The 1 mmol L⁻¹ solution of ligand was prepared by dissolving 0.0299 g of 3-[4-(dimethylamino)cinnamoyl]-4-hydroxy-6-methyl-2H-pyran-2-one in 100 mL of acetonitrile (Merck, Germany). The pH of the aqueous phase was adjusted by the addition of CH₃COOH-NH₄OH buffer solutions, which were prepared by mixing equimolar solutions (1 mol L⁻¹) of CH₃COOH and NH₄OH (both Centralchem, Slovakia) in various ratios (v/v). The buffer solution with pH 8 was prepared in ratio of 24.65:25.35 (CH₃COOH:NH₄OH, v/v).

2.2. Apparatus

A Lightwave II UV-vis spectrophotometer (Biochrom Ltd., United Kingdom) and a Specord S 600 diode-array spectrophotometer (Analytik Jena AG, Germany) equipped with matched

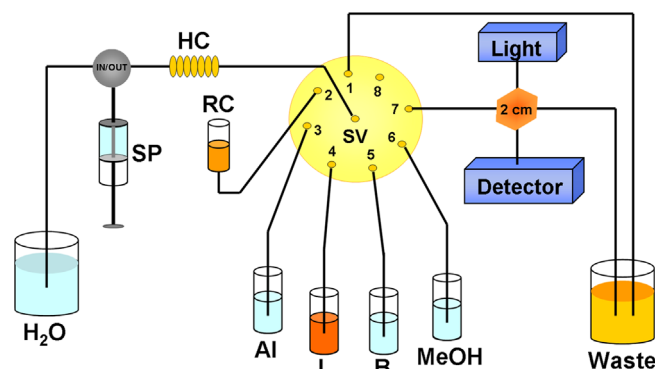


Fig. 2. The suggested set-up of sequential injection manifold. SP, syringe pump; HC, holding coil; RC, reaction chamber; SV, selection valve; L, ligand; B, buffer solution.

quartz cells of 10 mm path length were used for UV-vis spectrophotometric measurements. The pH values were measured using an ORION 720A⁺ pH metre with a glass electrode. The ATR measurement technique was performed on a Nicolet 6700 FT-IR spectrometer (Thermo Fisher Scientific Inc., USA).

The sequential injection manifold (Fig. 2) is based on the commercially available FIALab[®] 3500 system (FIALab[®] Instrument Systems Inc., Bellevue, USA) and equipped with a glass syringe pump (SP) (5 mL) and a central eight-port Cheminert selection valve (Valco Instrument Co., Houston, USA). The central port of the selection valve (SV) was connected to a holding coil (HC), and the lateral ports of the SV were connected as follows: waste (port 1), reaction chamber (RC – a 2 mL microcentrifuge polypropylene tube with a snap-on cap and 1.2 cm of i.d. width) (port 2), sample (Al) (port 3), ligand (port 4), buffer solution with pH 8 (port 5), methanol (port 6), air (port 8). Port 7 was directed to an optical Z-flow cell (20 mm) and flowed into a waste receptacle. An LS-1-LL tungsten halogen lamp (Ocean Optics Inc., Dunedin, USA) was used as the light source and the CCD USB 2000 diode array spectrophotometer (Ocean Optics Inc., Dunedin, USA) as the detector. FIALab[®] software (version 5.9.321) served as the operating programme, enabling the control of the flow procedure and the acquisition and evaluation of data.

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