Challenge

Trace element analysis of impurities in high-purity urea featuring complex emission spectra originating from the sample matrix.

Solution

HR ICP-OES with high-resolution optical system, superior sensitivity & matrix tolerance to achieve lowest detection limits for trace element analysis in urea.

Intended audience

QC & testing laboratories in urea production, AdBlue®/AUS 32 manufacturers, chemists performing trace element analysis, ICP-OES users in the chemical & fertilizer industries.

Detection of trace elements in urea by high resolution ICP-OES

Introduction

Urea is an organic compound that plays an important role in biological metabolism processes. Besides its physiological properties, synthetically synthesized urea has become a very meaningful compound in the clinical, pharmaceutical, and chemical industries. It is a starting material for the synthesis of a large number of chemicals including hydrazine and melamine that are further used for the production of resins eventually used as building materials. In agriculture, urea is a preferred compound to be used as fertilizer because of its high nitrogen content.

Another application with constantly rising importance is its use as an active ingredient in exhaust gas treatment processes such as SNCR and SCR for environmental protection purposes. In these processes, ammonia or urea is injected to the exhaust gases of power plants (incineration plants, gas turbines, etc.), utility vehicles, and automobiles to reduce the emission of NOX gases to the environment by reaction to harmless products such as water and nitrogen.

Due to its manifold application areas (chemistry, food and agriculture, environment, etc.), the quality of urea and its products is highly regulated. For instance, the quality of urea as NOX reduction agent AUS 32 in diesel engines is regulated by ISO 22241. Here, maximum limits for calcium, iron, copper, zinc, chromium, nickel, aluminum, magnesium, sodium, potassium, and phosphorus (as phosphate) are in the range of 0.2 to 0.5 mg/kg. Nevertheless, the demands for other application areas might be more stringent, and QC laboratories aim towards lower matrix-specific limits of detection for specification analysis.

The proposed method for trace element detection is by optical emission spectroscopy with inductively coupled plasma. For the product control of urea by ICP-OES, high precision and the lowest detection limits are desirable, yet the detectability of many trace elements is affected by signal suppression and drifting (matrix load) as well as by spectral interferences. The latter often require the use of less sensitive alternative lines, which often have lower sensitivity.

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When using the High-Resolution Array ICP-OES, many of these analytical shortcomings can be avoided.

Here, the exceptionally high spectral resolution and sensitivity of the PlasmaQuant 9200 Elite by Analytik Jena offers a new analytical potential! It allows for an interference-free analysis of trace elements in a complicated matrix such as urea. The high plasma robustness of the high-frequency generator, combined with the sample introduction system—the core component of which is the V-Shuttle Torch—enables exceptionally accurate and precise analysis, even for samples with high matrix interference.

Within this study, the PlasmaQuant 9200 Elite was evaluated regarding the matrix specific detection limits for twenty commonly tested trace elements. Furthermore, the analysis of a QC control standard was included for method validation and long-term stability testing.

Materials and Methods

Samples and reagents

A high-purity urea sample was analyzed. Multi-element and single-element stadards were used for the calibration and spiking experiments.

Sample preparation

For the analysis, a nitric acid solution with a urea concentration of 130 g/L was prepared. Aliquots of this solution were spiked with a 1 g/L multi-element and single-element standard to obtain a seven-point calibration curve.

Calibration

Table 1: Concentration of calibration standards

Element	Unit	Cal.0	Cal.1	Cal.2	Cal.3	Cal.4	Cal.5	Cal.6
Al, As, Ba, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, Pb, Si, Tl, Zn	µg/L	0	10	50	100	200	500	1,000
P	µg/L	0	100	500	1,000	5,000	10,000	–

Instrumentation

For the analysis, a PlasmaQuant 9200 Elite equipped with Salt-Kit and Teledyne CETAC ASX-560 autosampler was used. Representative test measurements on a PlasmaQuant 9200 Elite confirmed consistent performance under adjusted plasma conditions. The detailed system configuration is given in Table 2.

Table 2: Plasma configuration and set-up of the sample introduction system

Parameter	Specification
Power	1400 W
Plasma gas flow	9 L/min
Auxillary gas flow	0.8 L/min
Nebulizer gas flow	0.65 L/min
Nebulizer	Concentric nebulizer for high salt solutions, borosilicate, 2.0 mL/min
Spray chamber	Cyclonic spray chamber with dip tube, 50 mL, borosilicate
Outer tube/inner tube	Quartz/quartz
Injector	Quartz, 2 mm
Pump tubing	PVC
Sample pump rate	1.0 mL/min
Rinse/read delay	45 s
Autosampler	Teledyne CETAC ASX-560
Additional	Argon humidifier

Evaluation parameter

Table 3: Overview of method specific evaluation parameters

Element	Line [nm]	Plasma view	Integration mode	Read time [s]	Evaluation			
					No. of Pixel	Baseline fit	Polynomial degree	Correction
Al	396.152	axial	peak	3	3	ABC[1]	auto	–
Al	167.022	axial	peak	3	3	ABC	auto	–
As	188.979	axial	peak	10	3	ABC	auto	–
As	228.812	axial	peak	10	3	ABC	auto	–
Ba	455.403	axial	peak	3	3	ABC	auto	–
Ba	233.527	axial	peak	3	3	ABC	auto	–
Ca	396.847	axial	peak	3	3	ABC	auto	–
Ca	393.366	axial	peak	3	3	ABC	auto	–
Cd	214.441	axial	peak	3	3	ABC	auto	–
Cd	226.502	axial	peak	3	3	ABC	auto	–
Co	228.615	axial	peak	3	3	ABC	auto	–
Co	237.863	axial	peak	3	3	ABC	auto	–
Cr	205.552	axial	peak	3	3	ABC	auto	–
Cr	267.716	axial	peak	3	3	ABC	auto	–
Cu	324.754	axial	peak	3	3	ABC	auto	–
Fe	259.940	axial	peak	3	3	ABC	auto	–
Fe	238.204	axial	peak	3	3	ABC	auto	–
K	766.491	axial	peak	3	3	ABC	auto	–
K	769.897	axial	peak	3	3	ABC	auto	–
Mg	279.553	axial	peak	3	3	ABC	auto	–
Mg	285.213	axial	peak	3	3	ABC	auto	–
Mn	257.610	axial	peak	3	3	ABC	auto	–
Mn	259.372	axial	peak	3	3	ABC	auto	–
Mo	202.030	axial	peak	3	3	ABC	auto	–
Mo	281.615	axial	peak	3	3	ABC	auto	–
Na	589.592	radial	peak	3	3	ABC	auto	–
Na	588.995	radial	peak	3	3	static	auto	–
Ni	213.858	axial	peak	3	3	ABC	auto	–
Ni	221.648	axial	peak	3	3	ABC	auto	–
P	213.618	axial	peak	3	3	ABC	auto	–
P	214.914	axial	peak	3	3	ABC	auto	–
Pb	220.353	axial	peak	3	3	ABC	auto	–
Si	251.611	axial	peak	3	3	ABC	auto	–

Tl	190.796	axial	peak	3	3	ABC	auto	–
Zn	206.200	axial	peak	3	3	ABC	auto	–
Zn	213.856	axial	peak	3	3	ABC	auto	–

1 ... automatic baseline correction (ABC)

Results and Discussion

Using a high-resolution ICP-OES enables the interference-free analysis of trace elements as displayed in Table 4. Neither matrix based nor inter-element interferences were observed for the analysis of multiple lines of 20 typically measured trace elements. The investigation resulted in exceptional matrix-specific limits of detection in the low-ppb range (<20 µg/kg) for all investigated elements and lines. Method validation, including a QC recovery test of the Cal.3 standard after four hours of continuous measurement of a 130 g/L urea solution, demonstates the method's long-term stability, which is crucial for routine analysis. Recovery values between 97% and 104% were achieved.

Table 4: Overview of recovery values for CQ Cal.3 and achieved matrix specific limits of detection in 13% urea

Element	Line [nm]	Recovery QC Cal.3 [%]	Matrix specific LOD [mg/kg]
Al	396.152	100	0.010
Al	167.022	102	0.014
As	188.979	99.2	0.030
As	228.812	101	0.041
Ba	455.403	99.6	0.001
Ba	233.527	98.1	0.003
Ca	396.847	100	0.006
Ca	393.366	99.9	0.004
Cd	214.441	98.0	0.002
Cd	226.502	97.7	0.002
Co	228.615	97.2	0.004
Co	237.863	98.0	0.011
Cr	205.552	100	0.011
Cr	267.716	97.2	0.005
Cu	324.754	100	0.004
Cu	327.396	101	0.011
Fe	259.940	102	0.014
Fe	238.204	99.3	0.022
K	766.491	97.4	0.018
K	769.897	106	0.016
Mg	279.553	100	0.001
Mg	285.213	100	0.003
Mn	257.610	100	0.0004
Mn	259.372	102	0.001
Mo	202.030	99.5	0.013
Mo	281.615	101	0.009
Na	589.592	103	0.014
Na	588.995	104	0.024
Ni	213.858	98.4	0.005
Ni	221.648	102	0.008
P	213.618	99.8	0.092
P	214.914	96.5	0.140
Pb	220.353	102	0.032
Si	251.611	101	0.016
Si	288.158	98.1	0.037
Tl	190.796	97.3	0.041
Zn	206.200	97.7	0.003
Zn	213.856	99.4	0.004

Figure 1 shows representative spectra of selected elements, each with a reference line. For this, a 13% urea solution in 2% HNO_3 (v/v) was spiked with 100 µg/L of the respective element, or 1 mg/L for P. Several interference-free emission lines are available for each element. Due to the high spectral resolution, the most sensitive line can be specifically selected without interferences of other peaks. This is particularly evident for the P and As emission lines.

As an example, a correction for spectral interferences (CSI) was performed for the As line at 188 nm and the results are shown in Figure 2.

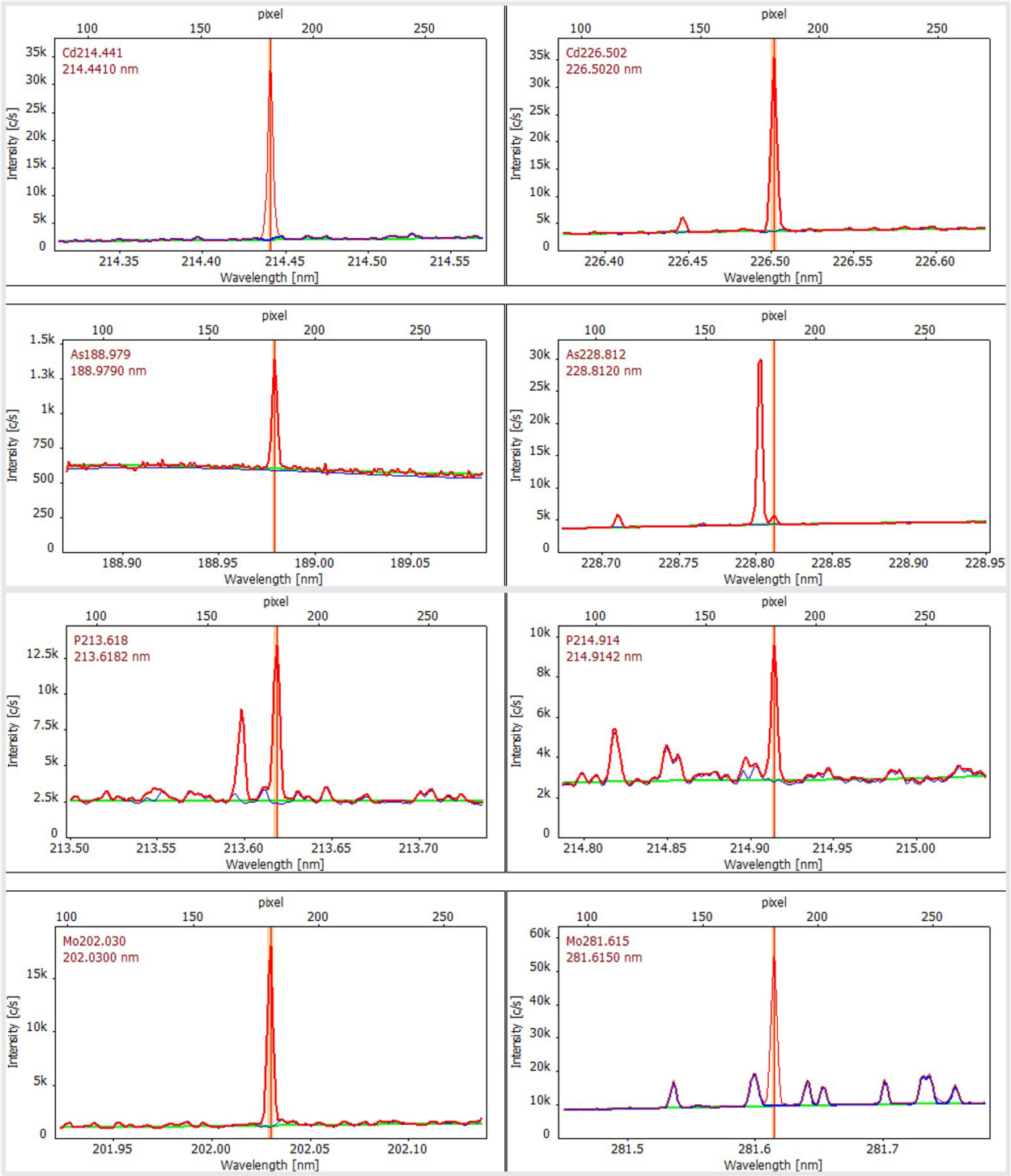

Figure 1: Sample spectra of selected lines

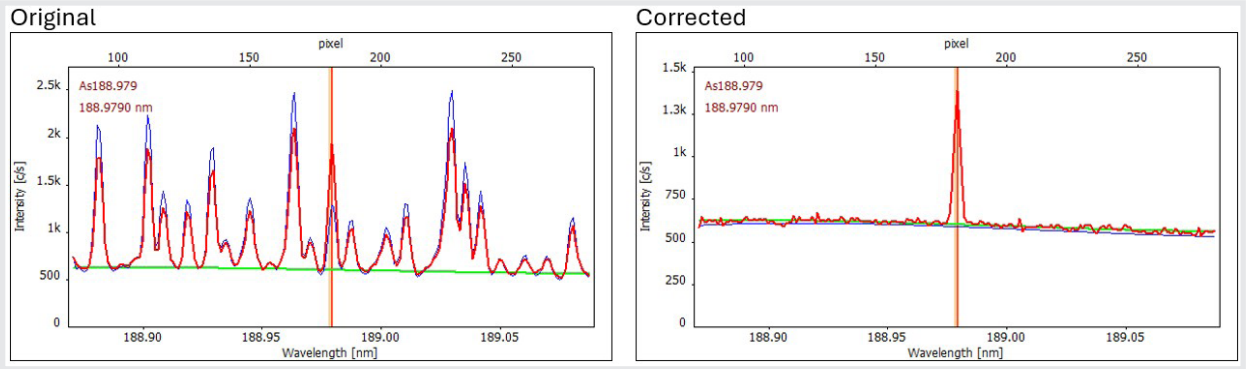

Figure 2: Sample spectra of selected lines

Summary

The yearly production of urea is amongst the highest of all synthetically produced chemicals, because of its versatile application in many industries. Urea contributes to food production as fertilizer, protects the environment as an ingredient of exhaust cleaning systems, and is used as starting material for the production of materials used every day. Therefore, the quality requirements for urea are high, which translates into a demanding task for quality-control labs on a daily basis.

While conventional ICP-OES systems struggle due to analytical challenges such as matrix bases spectral interferences and plasma robustness for matrix-rich samples, the PlasmaQuant 9200 Elite masters this demanding application without effort. Features such as the high-frequency generator and V-Shuttle torch allow for great plasma robustness in terms of short- and long-term stability. QC recovery values of 100% ± 4% are achieved after 4 hours of constantly running 13% urea samples. This high robustness allows for less dilution of the samples and therefore the possibility to reach lower limits of detection. In combination with the exceptionally high resolution, most sensitive lines can be used and compromises on line selection are not needed.

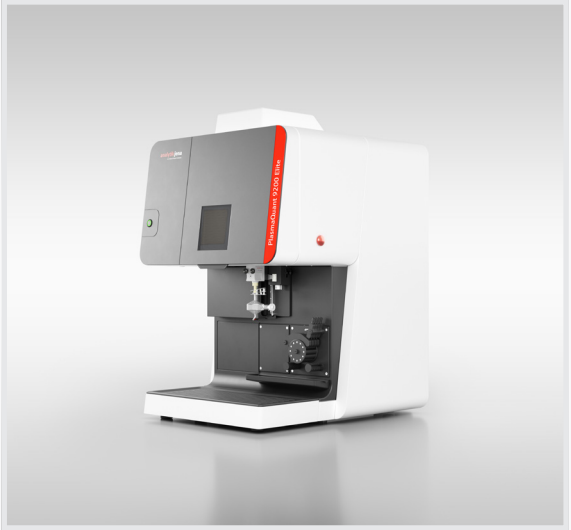

Figure 3: PlasmaQuant 9200 Elite

The PlasmaQuant 9200 Elite is able to achieve matrix dependent limits of detection which are about 100 times lower than the requirements of ISO 22241. The combination of high analytical performance and ease of use and data handling (automatic background correction) make the PlasmaQuant 9200 Elite the ideal tool for QC labs of the chemical industry.

Recommended device configuration

Table 5: Overview of devices, accessories, and consumables

Article	Article number	Description
Plasma Quant 9200 Elite	818-09201-2	High resolution ICP-OES
SALT KIT for PlasmaQuant 9x00 series	810-88561-0	Sample introduction kit for aqueous samples with high salt content (e.g. brine)
Teledyne CETAC ASX-560	810-88015-0	Autosampler with integrated rinse function

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