



Perkin Elmer 4100zl Manual

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Perkin Elmer 4100zl Manual:

Entwicklung und Anwendung eines direkten Verfahrens für die Analyse von Siliciumnitrid auf der Basis des Elektrothermischen Atomabsorption-Spektrometrie und der Suspensionstechnik Klaus-Christian Friese, 1997-06-13

Inhaltsangabe Zusammenfassung Auf der Grundlage der Slurry Technik d h durch Herstellung eines w rigen Suspension des feinpulvrigen Probenmaterials wurden Methoden f r die Bestimmung von Al Cr Cu Fe K Mg Na und Zn im Ultra Spurenbereich mittels Graphitrohr AAS entwickelt Das verwendete Spektrometer war ein Perkin Elmer 4100 ZL mit querbeheiztem Graphitrohrfen Durch Umgehung der Aufschlu prozedur wurde das Bestimmungsverfahren beschleunigt und gleichzeitig die Gefahr von Blindwerteintr gen massiv reduziert Dadurch wurde f r die Mehrzahl der Analytelemente eine erhebliche Verbesserung bis zu Faktor 100 des Nachweisverm gens erreicht Durch Einsatz eines chemischen Modifiers konnten Doppelpeaks bei der Atomisierung der Analytelemente eliminiert werden Die Richtigkeit der entwickelten Methode wurde anhand des Vergleichs mit Resultaten unabh ngiger Methoden berpr ft Inhaltsverzeichnis Inhaltsverzeichnis I Einf hrung und theoretische Grundlagen1 1 Siliciumnitrid1 1 1 Physikalische Eigenschaften1 1 2 Chemische Eigenschaften2 1 3 Herstellung3 1 4 Verwendung4 1 5 Analyse von Siliciumnitrid5 2 Atomabsorptionsspektrometrie10 2 1 Grundlagen10 2 2 Elektrothermische Atomabsorptionsspektrometrie11 2 3 Untergrund Kompensation12 2 4 Einsatz von Modifiern in der ETAAS15 2 5 Slurry Technik in der ETAAS17 3 Aufgabenstellung der Diplomarbeit20 II Experimenteller Teil22 1 Beschreibung der untersuchten Proben22 2 Verwendete Reagenzien und Ger te24 2 1 Aufschlu reagezien Modifier Standardl sungen24 2 2 Ger te24 3 Durchf hrung25 3 1 Aufschl sse25 3 2 Herstellung der Suspensionen26 3 3 Standardisierung27 3 4 AAS Messungen28 III Entwicklung und Anwendung eines Slurry ETAAS Verfahrens f r die Analyse von Siliciumnitrid30 1 Einsatz und Optimierung von Modifier30 1 1 Beschreibung der beobachteten St rungen30 1 2 Eliminierung der St rungen und Optim der chem Modifizierung32 1 3 Beobachtungen der Probenverteilung mittels Elektronenmikroskopie34 2 Nicht elementspezifische Untersuchungen43 2 1 Untersuchung des Verhaltens der Matrix mit der Radiotracerstechnik43 2 2 Radiotracerexperiment zur Adsorption von Analyten47 2 3 Stabilisierung von Analyt durch die Probenpulver48 2 4 Lebensdauer der Graphitrohre50 3 Optimierung des Analyseverfahrens f r die einzelnen Elemente52 3 1 Eisen52 3 2 Mangan57 3 3 Kupfer60 3 4 Chrom62 3 5 Natrium und

Entwicklung und Anwendung einer Suspensionstechnik für die Element-Analyse von Graphitpulver mit elektrothermischer Atomabsorptionsspektrometrie Uwe Schäffer, 1997-06-17

Inhaltsangabe Zusammenfassung Entsprechend den physikalischen und chemischen Eigenschaften von Graphit ist zu erwarten d a dieses Material nur schwer durch Aufschlu in L sung gebracht werden kann Verschiedene Versuche mit Ans tzen wie Perchlors ure Periods ure Salpeters ure Schwefels ure Salzs ure Salpeters ure und Kaliumdichromat Phosphors ure f hrten teils nicht zum vollst ndigen Aufschlu der Proben teils f hren die zeit und chemikalienaufwendigen Techniken zu hohen Blindwerten wodurch die Nachweisgrenzen der Bestimmungsmethoden

erheblich verschlechtert werden. In der vorliegenden Diplomarbeit wurden Druck und Mikrowellenaufschlüsse in Salpetersäure-Schwefelsäure-Gemischen durchgeführt, ohne dass die verwendeten Graphitproben vollständig in Lösung gebracht werden konnten. Bei der trockenen Veraschung von Graphitpulver im Elektroofen mit anschließendem Lösen in Säuren sind zum Teil Analytverluste bis 50 % z.B. bei Cr und K aufgetreten. Mit der Suspension GFAAS ist ein Verfahren gegeben, das vor allem als Ersatz für problematische Verbundverfahren eine Alternative mit bemerkenswerten Vorteilen darstellt. Die slurry sampling Technik beruht auf der Herstellung einer homogenen Suspension aus welcher ein Aliquot direkt in eine Atomisierungseinheit einpipettiert werden kann. Damit werden die Vorteile der direkten Methoden z.B. kein Aufschluss von der für Flüssigkeiten gebräuchlichen volumetrischen Dosierung erspart. Zusätzlich besteht die Möglichkeit, das Suspensionsmedium kurz vor Herstellung einer Suspension auf Vorhandensein eines Blindwertes zu untersuchen. Ansätze mit stark erhöhtem Blindwert werden verworfen. Als geeignetes Suspensionsmedium für Graphitpulver hat sich eine stark verdünnte Triton X 100 Lösung 0,008% bzw. zur Bestimmung von K eine 10%ige EtOH Lösung erwiesen. Für jedes Element wurden die optimalen Vorbehandlungs- und Atomisierungsbedingungen Zeit und Temperatur bestimmt. Ausgenommen Mg und Si konnten alle betrachteten Analytelemente ohne Einsatz von Modifier bestimmt werden. Die Vorteile, die sich daraus ergeben, sind kleinere Blindwerte infolge eines verringerten Kontaminationsrisikos und entsprechend bessere Nachweisgrenzen. Da bei der Bestimmung von Spurenelementen in Graphitpulver die Kalibrierung mit wässrigen Standardlösungen zu zufriedenstellenden Ergebnissen führte, kann auf die umständlichere Kalibrierung durch Aufstockung verzichtet werden. Dieses Zeitersparnis führt zusammen mit den kurzen

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