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文章摘要

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Determination of Silicon Manganese and Phosphorus in High Carbon-chrome Iron by Inductively Coupled Plasma-Atomic Emission Spectrometry after Microwave Digestion

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中文关键词： 高碳铬铁 硅 锰 磷 微波消解 电感耦合等离子体发射光谱法

英文关键词: high carbon-chrome iron silicon manganese phosphorus microwave digestion Inductively Coupled Plasma-Atomic Emission Spectrometry

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中文摘要:

高碳铬铁需要用强氧化性酸或混合酸在高温条件下才能分解或采用碱熔法处理样品，但操作比较繁琐并引入大量的钠离子，干扰待测元素的检测。微波消解技术与电感耦合等离子体光谱（ICP-AES）结合用于测定高碳铬中的铁已有报道。本文在文献方法的基础上，建立了ICP-AES同时测定高碳铬铁样品中Si、Mn、P的方法。采用硝酸消解高碳铬铁中易分解部分，再用高氯酸和氢氟酸消解碳化物和硅化物，减少了高氯酸的用量，消解过程平稳安全。利用高碳铬铁标准物质建立标准曲线，Y和In作内标，Si、Mn、P的检出限分别为0.0017%、0.0025%和0.0033%。用高碳铬铁标准物质GSB 03-1562—2003、GSB 03-1058—1999和GSB 03-1059—1999验证方法的精密度及准确度，11次测定的相对标准偏差（RSD）在0.8%~6.0%之间，不同含量标准物质测定结果与标准值吻合。本方法微波消解样品完全，操作安全简单，准确度高，适用于高碳铬铁样品的多元素同时分析。

英文摘要:

High carbon-chrome iron samples can be decomposed using strong oxidizing acid, mixed acid or by the alkali fusion method under high temperature. These chemical procedures are complex, especially the alkali fusion method, which introduces a large amount of interfering ions such as Na^{+}. Microwave digestion technology is widely used in the pretreatment process of insoluble samples. This pretreatment method combined with Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES) has been reported to determine Fe in high carbon-chrome iron. Based on previous studies, a method for the simultaneous determination of silicon, manganese and phosphorus has been established in this study. HNO_3 is conducted to digest easy decomposition components in high carbon-chrome iron. $HClO_4$ and HF are then loaded to digest carbides and silicides. Since there is a reduction in the volume of $HClO_4$, the process is stable and safe. Using high carbon-chrome iron standard materials to set up the calibration curve, and Y, In and Bi as internal standard elements, the detection limits of silicon manganese and phosphorus were 0.0017%, 0.0025% and 0.0033%, respectively. Precision and accuracy of this method were verified by using high carbon-chrome iron standard materials of GSB 03-1562—2003, GSB 03-1058—1999 and GSB 03-1059—1999. The results were consistent with the certified values. The relative standard deviations (RSD) are in the range 0.8%-6.0% (n=11), and the relative deviations (RE) are from 0.044% to 0.227%. This method has the advantage of digesting the sample completely, is safe and has a simple operation process and a high degree of accuracy, which can also be used for the determination of several elements in high carbon-chrome iron.

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