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Development of Analytical Methods for Ultra-trace Elements in High Purity Iron and Steel

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:

MIBK
Al Mn Mo W 30
Si P B

ICP-MS Sb Pb Si P B
/g

Synopsis :
Analytical methods were developed to improve the sensitivity of detecting ultra-trace elements in high purity iron and steels. A method to decompose a sample in a single test tube was developed for the decrease of contamination and convenient sample preparation. Matrix Fe was eliminated by MIBK extraction and anion exchange chromatographic separation for the concentration of analytes and the removal of the interference of matrix on the measurement by ICP-MS. More than thirty elements including Al, Mn, Mo, etc. in high purity iron and steels were able to be determined using these two methods of sample preparation by ICP-MS. Because trace Si, P, and B could not be determined using the above-mentioned methods, the gel chromatographic separation for trace Si and P analysis and the ion exchange chromatographic separation for trace B analysis were developed respectively. The limits of determination were

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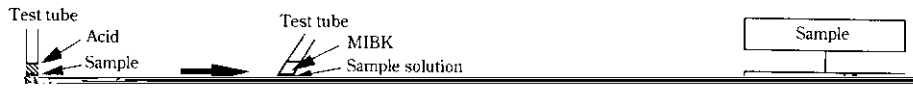
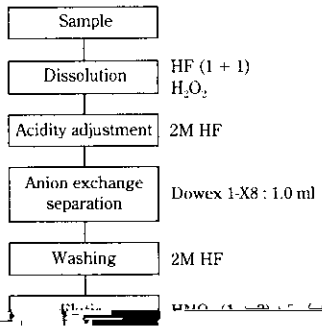
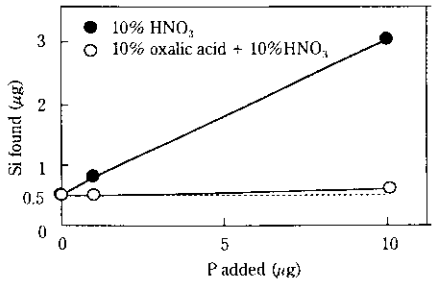
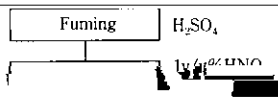

Test tube
Acid
Sample
Test tube
MIBK
Sample solution
Sample


Sample
Dissolution
HF (1 + 1)
H_2O_2
Acidity adjustment
2M HF
Anion exchange separation
Dowex 1-X8 : 1.0 ml
Washing
2M HF


10% HNO_3
10% oxalic acid + 10%HNO_3
Si found (μg)
P added (μg)
0
0.5
1
2
3
5
10


Fuming
H_2SO_4

6 結　言

マトリックスを除去した。さらに，Cr を含有する鋼試料については CrO_2Cl_2 揮散により Cr を除去した。

オキソ酸形成元素の分析については，目的元素をふっ化物錯

した。

(1) 試料溶液調製方法

試験管分解法を開発し，汚染の低減と分析操作の迅速・簡便

吸着分離法で，B 分析は，イオン交換分離法でマトリックスを分離した。

(3) 定量下限