

Table II Mean Absolute Variations in Assay Values for Thirteen of the Added Elements	
Technique	Average differences, ppm
AA (1)	7.5
ICPES (2)	13.7
DRES (3)	21.7
PHES (4)	16.6

overall mean was 3 ppm which is much more reasonable. As expected, silicon was picked up from the trays used for the sponge reduction stage.

The high palladium result is unlikely to be due to palladium in the starting sponge. A possible explanation may be that an error occurred during the preparation, resulting in a true content of 125 ppm. For most of the remaining elements the results are in close agreement with the true content of 100 ppm. The exceptions are antimony and arsenic, where some volatilisation during the reduction stage was expected even though the temperature was kept as low as practicable.

Summary of the Results and Conclusions

On the evidence of the results, and providing that sufficient care is taken, AA should be preferred for standardisation assays on non-synthetic standard materials, since the values found agree well with the true contents and the variability is the lowest for the four methods. This may be visualised independently by summing the absolute differences from the standard value for all the elements. For thirteen of the added elements, and with the value for palladium taken as 125 ppm, the average differences for the four techniques are given in Table II. The advantages of ICPES are speed and, for some of the elements, sensitivity, but these may have been offset by the complex effects of the high platinum loadings which can

directly affect sensitivity and necessitate careful background correction.

The variability of the results both between laboratories and within laboratories when using different analysis methods has emphasised the need for standards of the type produced. The spread of results also means that analytical differences of less than 10 ppm (10 per cent relative) between samples may have no significance, unless large numbers of samples are analysed.

The successful method of preparation used for platinum is generally applicable, and is currently being used for some of the other platinum group metals.

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Palladium in a Modified Steel

The degradation induced by the presence of hydrogen in some steels may be reduced or eliminated by the addition of palladium, but the mechanism for this effect is not fully understood. Little is known about the distribution of the palladium in such modified steels so a recently published study by M. K. Miller, of the Oak Ridge National Laboratory, and S. S. Brenner and M. G. Burke, of the University of Pittsburg, used high resolution atom probe field-ion microscopy and conventional transmission electron microscopy to examine the microstructure and microchemistry of laboratory melted low carbon alloy steels containing 0.31 and 0.65 atomic per cent palladium (*Metall. Trans.A*, 1987, **18A**, (4), 519-523).

The authors concluded that the solubility of palladium in the tempered steel was between 0.25 and 0.5 atomic per cent, considerably less than indicated by the iron-palladium phase diagram. Although most of the palladium remained in solution in the ferrite, some had segregated to the ferrite-ferrite lath boundaries where it formed a mono-atomically thick adsorbate layer. Palladium-manganese precipitates were detected both at lath boundaries and in the ferrite matrix.