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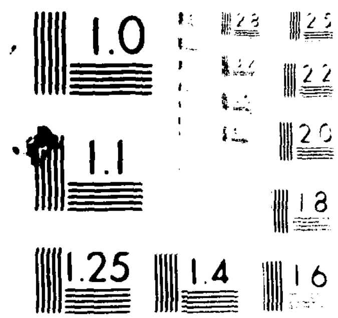
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Previously reported interferences observed with various oil standards are studied as a function of height.

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TECHNICAL REPORT NO. 29

The Role of Observation Height in Some Interferences Reported in Oil Analysis by Atomic Absorption Spectrophotometry

by

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Prepared for Publication
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The Role of Observation Height in Some
Interferences Reported in Oil Analysis by
Atomic Absorption Spectrophotometry

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Reports have appeared (1, 2) which indicate that the type of organometallic complex used in the preparation of standards for the determination of metals in oil by atomic absorption spectrophotometry (AAS) can influence the sensitivity of the method. Because the type of complexes existing in a given oil sample may not be known, this represents a possible interference on the determination.

Recent work in this laboratory (3) has shown that similar interferences can exist in the inductively coupled plasma (ICP), if observations are carried out near the load coil. The interferences were not seen when observations were made high in the plasma discharge.

The reported AAS interferences (1, 2) were observed very low in the flame, just above the burner. In contradiction to usual experience (4), the magnitudes of the reported interferences were greater in the nitrous oxide-acetylene flame than in the air-acetylene flame. Since observation height was found to affect the ICP results, an investigation of the role of observation height upon the reported interference on nickel absorption, as well as its effect on iron absorption was made.

Nickel 2,4-pentanedionate (nickel acetylacetonate, NiAA) nickel cyclohexanebutynate (NiCHB), and iron 2,4-pentanedionate (FeAA) were obtained from the Ventron Division of Thiokol (Danvers, MA). Nickel and iron standards were purchased from the Conostan Division of CONOCO, Inc. (Ponca City, OK) (NiCON, FeCON).

Standards containing 100 µg/ml of metal were prepared from the compounds by dissolving the appropriate amounts in 10 ml of pyridine and diluting them to 100 ml with mixed xylenes.

The absorbances of the resulting solutions were measured using a model AA-6 spectrophotometer (Varian, Palo Alto, CA). For iron, a wavelength of 248.3 nm was used with a 0.1 nm bandpass, while 341.5 nm and a 0.2 nm bandpass were used for nickel. An air-acetylene flame was used, with the acetylene flow reduced as far as possible when no sample was being aspirated. This gave a slightly fuel-rich appearance to the flame when xylenes were nebulized.

For iron, a slight interference was seen at an observation height of 5 mm above the burner, with FeAA giving a 3% higher absorbance signal than FeCON. This interference decreased with increasing observation height, and both FeAA and FeCON gave the same signal (see Figure 1) 18 mm above the burner.

The sensitivity varied with observation height as shown in Figure 2. The best sensitivity, 63 ppm/absorbance unit (ppm/AU), was obtained at 8 mm above the burner; however, the degradation associated with raising the observation height to 18 mm was not serious.

NiAA and NiCON gave equal absorbance values from 5 to 18 mm above the burner; however, a difference was seen between NiCHB and NiCON at 5 mm, with the NiCHB producing only 91% as much absorbance as did the NiCON. This effect was much reduced with increasing burner height, the NiCHB signal being 98% of the NiCON signal at an 18 mm observation height. The sensitivity for NiCON was 68 ppm/AU at 5 mm, degrading slightly to 73 ppm/AU at 18 mm.

These data suggest that observing well up in the flame alleviates the reported interference problem.

ACKNOWLEDGEMENT

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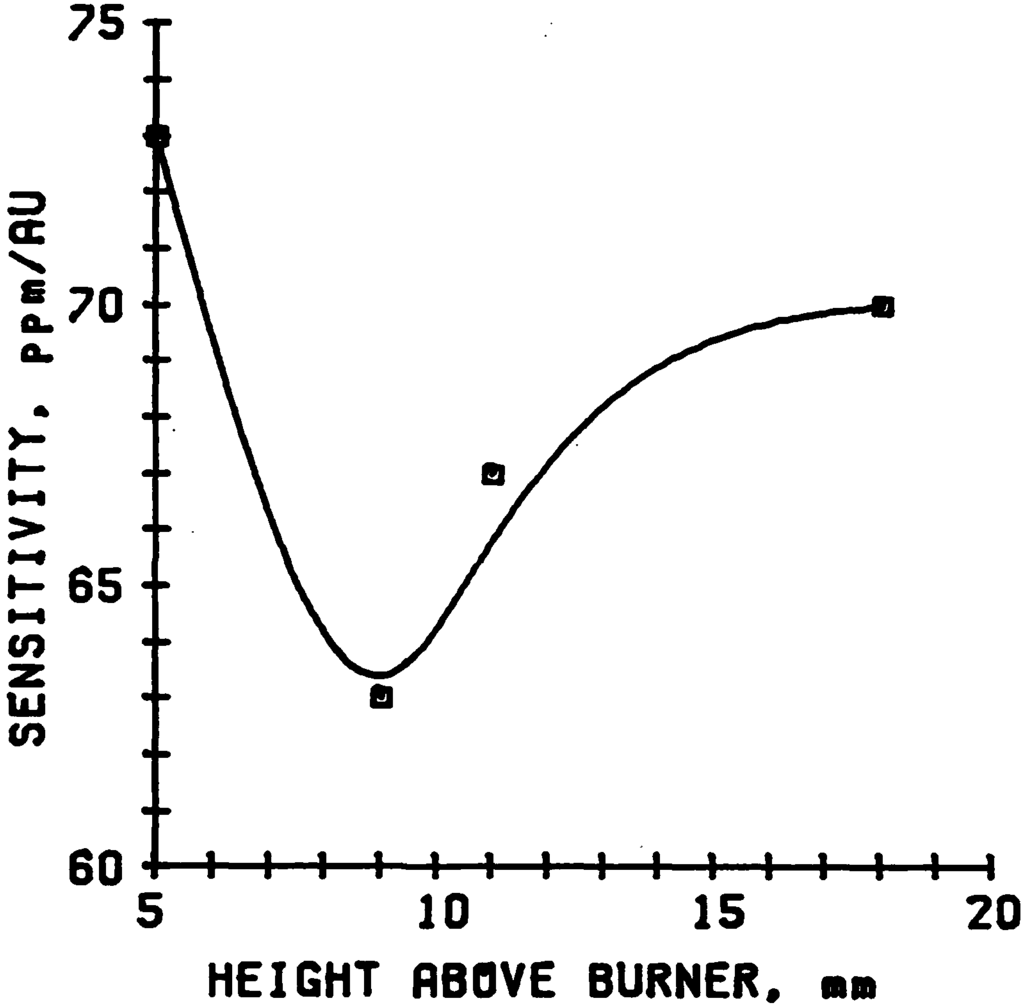
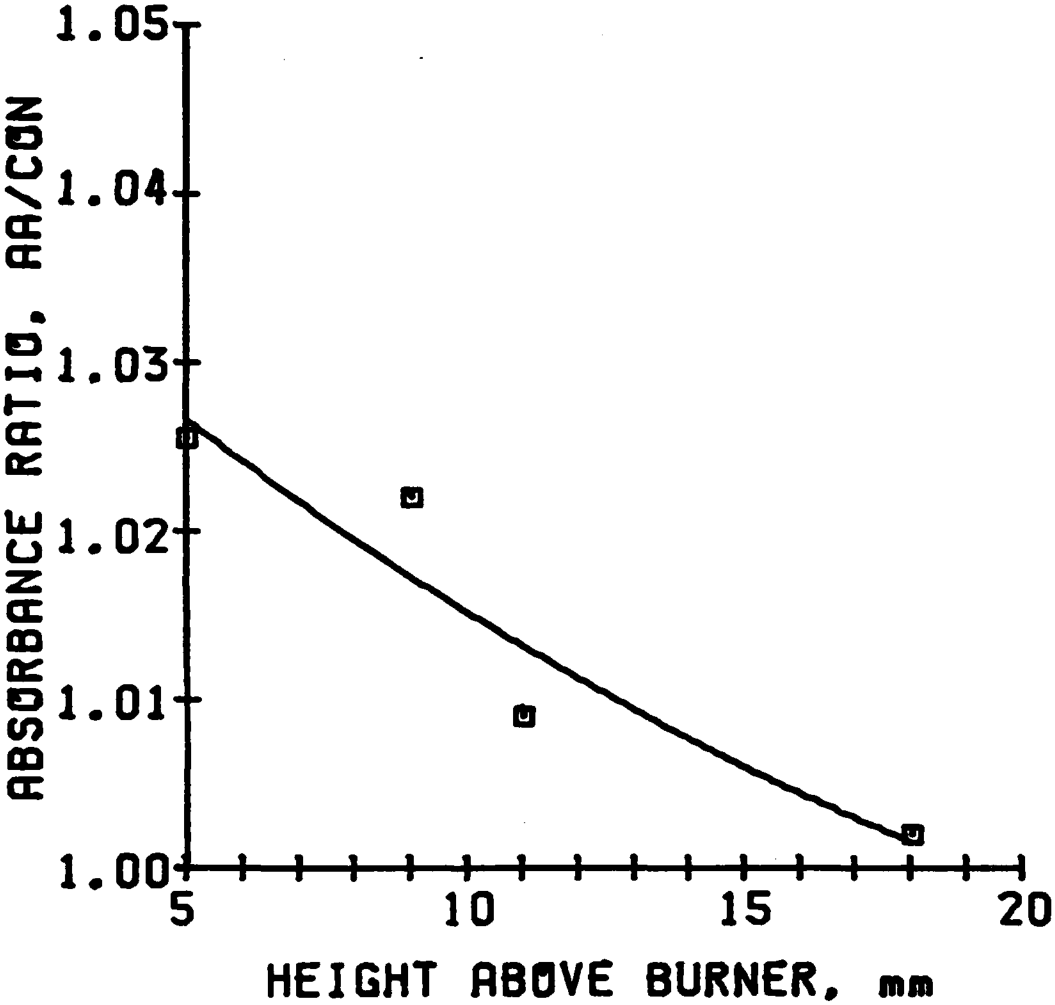
FIGURE CAPTIONS

Figure 1 Ratio of the Absorbance given by FeAA to that given by FeCON as a Function of Observation Height.

Figure 2 Sensitivity for Iron Analysis in ppm/Absorbance Unit as a Function of Observation Height.


75
70
65
60
5
10
15
20
SENSITIVITY, ppm/AU
HEIGHT ABOVE BURNER, mm



1.05
1.04
1.03
1.02
1.01
1.00
5
10
15
20
ABSORBANCE RATIO, AA/CON
HEIGHT ABOVE BURNER, mm