

Withdrawn
Provided for Historical Reference Only

characteristic mass value was 0.41 pg, calculated from the average peak area (abs-sec) reading of the 5 ng/mL standard which is approximately midway in the linear portion of the working standard range. This agreed within 20% with the characteristic mass value, 0.35 pg, listed by the manufacturer of the instrument (5.2.).

4.7.2. The peak area (abs-sec) readings of the reagent blank and the low concentration Cd standards from 0.1 to 2.0 ng/mL and statistical analysis of the results are shown in Table III. Five of the reagent blank peak area readings were zero and the sixth reading was 1 and was an outlier. The near lack of a blank signal does not satisfy a strict interpretation of the IUPAC method for determining the detection limits. Therefore, the standard deviation of the six peak area readings of the 0.2 ng/mL cadmium standard, 0.75 abs-sec, was used to calculate the detection limits by the IUPAC method. The slope, m , as calculated by a linear regression plot of the peak area (abs-sec) readings (shown in Table IV) of the 0.2 to 10 ng/mL cadmium standards versus their concentration is 51.5 abs-sec/(ng/mL).

4.7.3. If 0.75 abs-sec (sd) and 51.5 abs-sec/(ng/mL) (m) are used in Equation (4.7.3), the qualitative and quantitative detection limits as determined by the IUPAC method are:

$$C_{id} = (3)(0.75 \text{ abs-sec}) / (51.5 \text{ abs-sec}/(\text{ng/mL})) \\ = 0.044 \text{ ng/mL for the qualitative detection limit.}$$

$$C_{id} = (10)(0.75 \text{ abs-sec}) / (51.5 \text{ abs-sec}/(\text{ng/mL})) \\ = 0.15 \text{ ng/mL for the quantitative detection limit.}$$

The qualitative and quantitative detection limits for the AAS-HGA analytical technique are 0.44 ng and 1.5 ng cadmium, respectively, for a 1 mL solution volume. These correspond, respectively, to 0.007 $\mu\text{g}/\text{m}^3$ and 0.025 $\mu\text{g}/\text{m}^3$ for a 60 L air volume.

4.7.4. The peak area (abs-sec) readings of the Cd standards from 0.2 to 40 ng/mL and the statistical analysis of the results are given in Table IV. The recommended standard working range for AAS-HGA analysis is 0.2 to 20 ng/mL. The standard of lowest concentration in the recommended working range is slightly greater than the calculated quantitative detection limit 0.15 ng/mL. The deviation from linearity of the peak area readings of the 20 ng/mL standard, the highest concentration standard in the recommended working range, is approximately 10%. The deviations from linearity of the peak area readings of the 10 and 40 ng/mL standards are significantly greater than 10%. As shown in Table IV, the precision of the peak area readings are satisfactory throughout the recommended working range, the relative standard deviations of the readings range from 0.025 to 0.083.

4.8. Analytical Method Recovery for Flame AAS Analysis

4.8.1. Four sets of spiked MUEF samples were prepared by injecting 20 μL of 10, 50, 100 and 200 $\mu\text{g}/\text{mL}$ dilute cadmium stock solutions on 37 mm diameter filters (part no. AAWP 037 01, Millipore Corp., Bedford, MA) with a calibrated micropipet. The dilute stock solutions were prepared by making appropriate serial dilutions of a commercially available 1,000 $\mu\text{g}/\text{mL}$ cadmium standard stock solution (RICCA Chemical Co., Lot# A102) with the diluting solution (8% HNO_3 , 0.4% HCl). Each set contained six samples and a sample blank. The amount of cadmium in the prepared sets were equivalent to 0.1, 0.5, 1.0 and 2.0 times the TWA PEL target concentration of 5 $\mu\text{g}/\text{m}^3$ for a 400 L air volume.

4.8.2. The air-dried spiked filters were digested and analyzed for their cadmium content by flame atomic absorption spectroscopy (AAS) following the procedure described in Section 3. The 0.02 to 2.0 $\mu\text{g}/\text{mL}$ cadmium standards (the suggested working range) were used in the analysis of the spiked filters.

4.8.3. The results of the analysis are given in Table V. One result at 0.5 times the TWA PEL target concentration was an outlier and was excluded from statistical analysis. Experimental justification for rejecting it is that the outlier value was probably due to a spiking error. The coefficients of variation for the three test levels at 0.5 to 2.0 times the TWA PEL target concentration passed the Bartlett's test and were pooled.

4.8.4. The average recovery of the six spiked filter samples at 0.1 times the TWA PEL target concentration was 118.2% with a coefficient of variation (CV_1) of 0.128. The average recovery of the spiked filter samples in the range of 0.5 to 2.0 times the TWA target concentration was 104.0% with a pooled coefficient of variation (CV_1) of 0.010.

Note: OSHA no longer uses or supports this method (December 2019).

