

분석자료 처리

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I. 검출한계 및 정량한계의 의미

작업환경측정시료의 경우 대부분은 NIOSH나 OSHA의 측정분석방법을 참고하고 있는 것으로 알고 있다. 일정 수준의 교육과 실습과정을 거치면 정해진 절차에 따라 표준시료를 이용한 검량선작성, 시료 전처리, 기기분석, 결과 계산의 과정을 그리 어렵지 않게 따라갈 수는 있을 것이다. 그런데 이렇게 절차나 표준에 따라 실험을 하는 것도 중요하지만 여기에 더해 반드시 고려해야 할 점은 자기가 생산한 분석결과가 어느정도 신뢰성을 가지고 분석한 것인지를 설명할 수 있어야 한다는 것이다. 동일한 측정, 분석방법을 참고하여 분석을 하였더라도 기관마다 보유하고 있는 장비가 다르거나 그 상태가 다르며, 분석자의 숙련정도에도 차이가 있기 때문에 그 분석의 수준은 분석기관이나 분석자에 따라 많은 차이가 있을 수 있다. 예로써 NIOSH의 측정분석방법에서 제시한 검출한계가 0.01 mg/sample이라고 할지라도 어떤 기관은 1 mg/sample의 수준밖에는 검출할 수 없는 곳도 있고 어떤 기관은 0.01 mg/sample 보다 더 낮은 수준까지도 검출할 수도 있다. 혹자는 노출기준에 비해 많이 낮으면 되지 검출한계, 정량한계가 무엇이 중요한가라고 예기하는 경우도 있다. 그런데 낮은 농도수준까지 분석을 할 수 있다는 것은 작업환경의 농도를 그만큼 정확히 분석할 수도 있다는 얘기가 될 수도 있다. 예로써 STEL이나 ceiling과 같이 짧은 동안 측정한 시료의 경우는 분석가능한 농도수준에 따라 많은 영향을 받는다. 분석기기의 노후, 분석자의 숙련도가 낮아 정량 가능한 농도 수준이 상대적으로 높을 경우 사업장의 유해물질 노출농도가 노출기준과 비교할 때 작업환경 관리가 필요한 농도 이상임에도 불구하고 이를 정량한계 이하로 밖에는 평가할 수 없는 경우가 발생할 수도 있다. 따라서 분석가능한 농도수준에 대한 정확한 평가 없이 검출한계 또는 정량한계 이하라고 해서 측정시료 분석결과를 막연히 "ND", "trace"라고만 평가하는 것은 너무나도 막연한 결과를 생산한 것이다. 그리고 특정 공정이나 지역의 평균 노출농도를 구함에 있어서도 검출한계, 정량한계를 모르고서는 정확한 통계분석 결과를 얻기가 어렵다. 그리고

비용을 들여 작업환경측정을 한 사업주의 경우도 자신의 사업장의 농도가 노출기준과 비교하여 정확히 어느정도의 수준이라는 것을 알고 싶어할 것이고 측정기관은 이에 대한 정확한 수준을 알려주어야 할 의무가 있는 것이다. 이러한 이유로 분석결과에 대한 설명을 위해서는 검출한계와 정량한계를 거론하지 않을 수 없다. 시료분석에 대해 어느정도 경험이 있는 사람이라면 검출한계와 정량한계에 관해 한번쯤은 고민해 보셨을 것이라 생각한다. 동 교육이 여러분의 측정분석결과의 신뢰성을 향상하는데에 도움이 되기를 바란다.

II. 검출한계 및 정량한계 평가방법

OSHA, NIOSH, HSE 등에서 제공하고 있는 측정, 분석방법이나 측정 및 분석 평가지침을 참고하면 많은 도움이 된다. 여기서는 각 기관마다 정의하고 있는 검출한계, 정량한계의 개념과 어떻게 이를 평가하는지를 사례를 들어 설명하고자 한다.

1. 미국 OSHA의 검출한계, 정량한계 평가 및 사례

가. Determination of detection limits

Detection limits (DL), in general, are defined as the amount (or concentration) of analyte that gives an instrument response(Y_{DL}) that is significantly different (three standard deviations, SD_{BR}) from the background response(Y_{BR}).

$$Y_{DL} - Y_{BR} = 3(SD_{BR})$$

The direct measurement of Y_{BR} and SD_{BR} in chromatographic methods is typically inconvenient and difficult because Y_{BR} is usually extremely low. Estimates of these parameters can be made with data obtained from the analysis of a series of analytical standards or samples whose responses are in the vicinity of the background response. The regression curve obtained for a plot of instrument response versus concentration of analyte will usually be linear. Assuming SD_{BR} and the precision of data about the

curve are similar, the standard error of estimate (SEE) for the regression curve can be substituted for SD_{BR} in the above equation.

The following calculations derive a formula for DL:

$$SEE = \sqrt{\frac{\sum (Y_{obs} - Y_{est})^2}{n - k}}$$

Y_{obs} = observed response

Y_{est} = estimated response from regression curve

n = total no. of data points

k = 2 for a linear regression curve

At point Y_{DL} on the regression curve

$$DL = (Y_{DL} - Y_{BR}) / A \quad A = \text{analytical sensitive(slpoe)}$$

therefore

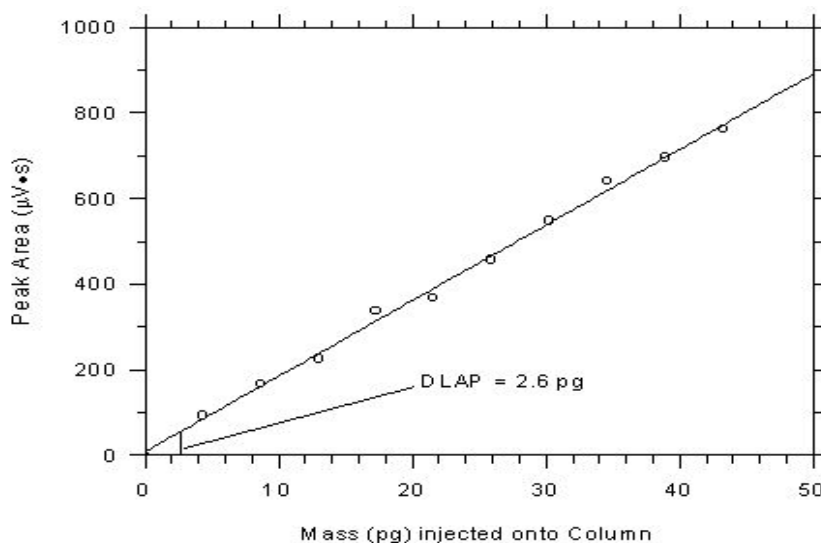
Substituting $3(SEE) + Y_{BR}$ for Y_{DL} gives

$$DL = 3(SEE) / A$$

4. Detection limit of analytical procedure(PLAP)

The DLAP is measured as the mass of analyte introduced into the chromatographic column. Ten analytical standards were prepared in equal descending increments with the highest standard containing 4325 ng of toluene per mL. This standard produces a peak approximately 10 times the baseline noise of a reagent blank when a 1- μ L injection with a 1:100 split is made onto the GC column. Standards, plus a reagent blank, were analyzed and the data obtained were used to determine the required parameters (A and SEE) for the calculation of the DLAP. Values of 17.66 and 15.3 were obtained for A and SEE respectively. The DLAP was calculated to be 2.60 pg.

□ 대상물질의 기기상의 분석가능 농도					
		ng/mL	pg	area	
		0	0	0	
1 uL injection		432.5	4.325	92.7	
split ratio 100 : 1		865	8.65	167	
		1298	12.98	226	
		1730	17.3	338	
		2162	21.62	368	
		2595	25.95	456	
		3028	30.28	547	
		3460	34.6	641	
		3892	38.92	696	
		4325	43.25	761	
회귀분석 통계량					
다중 상관계수	0.9983711				
결정계수	0.9967448		3표준오차	45.77	
조정된 결정계수	0.9963831		기울기	17.66	
표준 오차	15.2571		LOD	2.59	pg
관측수	11				
분산 분석					
	자유도	제곱합	제곱 평균	F 비	유의한 F
회귀	1	641487.61	641487.6	2755.778	1.6595E-12
잔차	9	2095.0124	232.7792		
계	10	643582.63			
	계수	표준 오차	t 통계량	P-값	
Y 절편	8.4013874	8.6064245	0.976176	0.354484	
X 1	17.65753	0.3363627	52.4955	1.66E-12	



다. Detection limit of the overall procedure (DLOP)

The DLOP is measured as mass per sample and expressed as equivalent air concentrations, based on the recommended sampling parameters. Ten samplers of each type were spiked with equal descending increments of toluene such that the highest sampler loadings were 4325 ng/sample for the adsorbent tubes and 8650 ng/sample for the 3M 3520 OVMs and SKC samplers. (The diffusive samplers were spiked with twice the amounts of toluene compared to adsorbent tubes because they are desorbed with 2 mL of solvent versus 1 mL for the adsorbent tubes.) These are the amounts, when spiked on the samplers, that would produce peaks approximately 10 times the baseline noise for sample blanks. These spiked samplers, plus blanks, were analyzed with the recommended analytical parameters, and the data obtained used to calculate the required parameters (A and SEE) for the calculation of the DLOPs. Values of 0.1768 and 14.5, 0.1745 and 20.0, 0.0831 and 18.2, and 0.0943 and 28.4 were obtained for A and SEE for charcoal tubes, Anasorb[®] 747 tubes, 3M 3520 OVMs and SKC 575-002 samplers respectively. The DLOPs were calculated to be 246 ng per sample (5.4 ppb or 20.5 µg/m³), 344 ng per sample (7.6 ppb or 28.7 µg/m³), 657 ng

per sample (25 ppb or 93 $\mu\text{g}/\text{m}^3$) and 904 ng per sample (67 ppb or 253 $\mu\text{g}/\text{m}^3$) for charcoal tubes, Anasorb[®] 747 tubes, 3M 3520 OVMs and SKC 575-002 samplers respectively.

□ 시료분석에 대한 종합적인 LOD 및 RQL 평가					
		ng/sample	area		
		0	0		
		432.5	90.1		
		865	162		
		1298	244		
		1730	322		
		2162	386		
		2595	491		
		3028	518		
		3460	639		
		3892	686		
		4325	776		
회귀분석 통계량					
다중 상관계수	0.9985274				
결정 계수	0.9970571		3표준오차	43.6	
조정된 결정 계수	0.9967301		기울기	0.1768	
표준 오차	14.524169		LOD	246.4 ng	
관측수	11		LOQ	821.4 ng	
			orRQL	68.5 $\mu\text{g}/\text{m}^3$	
분산 분석					
	자유도	제곱합	제곱 평균	F 비	유의한 F
회귀	1	643226.65	643226.6	3049.169	1.0539E-12
잔차	9	1898.5632	210.9515		
계	10	645125.21			
	계수	표준 오차	t 통계량	P-값	하위 95%
Y 절편	9.8296151	8.1929819	1.19976	0.260862	-8.7042116
X 1	0.1768145	0.003202	55.21928	1.05E-12	0.16957094

라. Reliable quantitation limit (RQL)

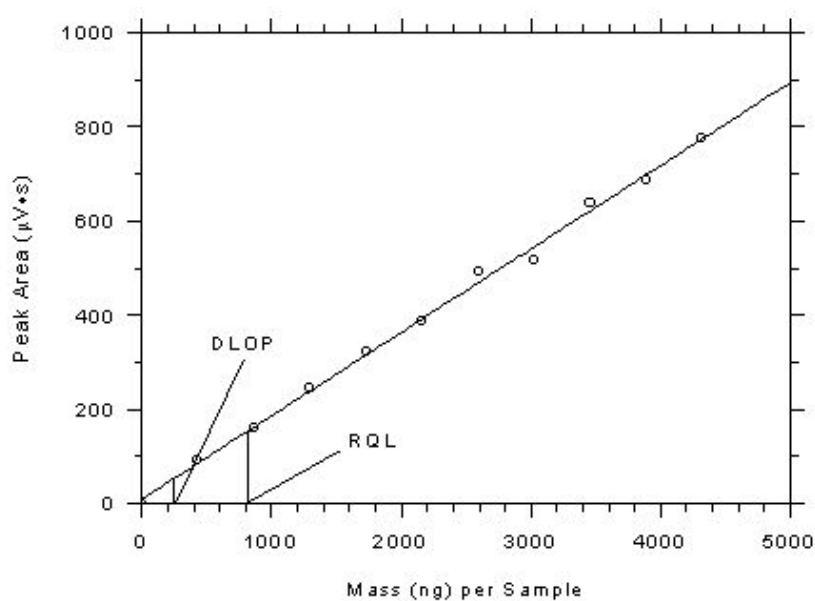
The RQL is considered the lower limit for precise quantitative measurements. It is determined from the regression line data obtained for the calculation of the DLOP). The RQL is defined as the amount of analyte that gives an instrument response (Y_{RQL}) such that

$$Y_{RQL} - Y_{BR} = 10(SD_{BR})$$

therefore

$$RQL = 10(SEE) / A$$

The RQLs were calculated to be 820 ng per sample (18.1 ppb or 68.3 $\mu\text{g}/\text{m}^3$).



※ NIOSH NMAM에서의 LOD

TABLE 4. MEASUREMENT RANGE AND PRECISION^a

Substance	LOD (µg/sample)	Measurement	
		Range (mg)	Precision (S _r)
benzene	0.5	0.004-0.35	0.013
p-tert-butyltoluene	1.1	0.013-1.09	0.017 ^a
cumene	0.6	0.039-3.46	0.017
ethylbenzene	0.5	0.045-8.67	0.015
α-methylstyrene	0.6	0.036-3.57	0.014
β-methylstyrene	0.6	0.036-0.728	0.014
toluene	0.7	0.024-4.51	0.022
o-xylene	0.8	0.044-10.4	0.014
m-xylene	0.8	0.043-0.864	0.013
p-xylene	0.7	0.043-0.861	0.015
styrene	0.4	0.181-8.49	0.014

바. Precision (overall procedure)

The precision of the overall procedure is determined from the storage data. The determination of the standard error of estimate (SEE_R) for a regression line plotted through the graphed storage data allows the inclusion of storage time as one of the factors affecting overall precision. The SEE_R is similar to the standard deviation, except it is a measure of dispersion of data about a regression line instead of about a mean. It is determined with the following equation:

$$SEE_R = \sqrt{\frac{\sum (Y_{obs} - Y_{est})^2}{n - k}}$$

n= total no. of data points

k= 2 for linear regression

k= 3 for quadratic regression

Y_{obs}= observed % recovery at a given time

Y_{est}= estimated % recovery from the regression line at the same given time

The following formula is used to determine the total standard error of estimate (SEE) for adsorbent tubes. An additional 5% for pump error (SP) is added to the SEE_R by the addition of variances.

$$SEE = \sqrt{(SEE_R)^2 + (SP)^2}$$

The following formula is used to determine the total standard error of estimate (SEE) for diffusive samplers when the sampling site temperature and pressure are known. SR is the sampling rate variability that has been determined in separate studies to be 6.4% for 3M OVMs and 8.7% for SKC samplers.

$$SEE = \sqrt{(SEE_R)^2 + (SR)^2}$$

The following formula is used when the sampling site temperature or pressure are not known. Additional variances are included that account for uncertainty for uncorrected temperature (ST) and pressure (SPr) effects on the sampling rate. When the temperature is unknown, it will be assumed that it is 22.2°C (295.4 K, 72°F) and a value of 7.7% shall be used for ST. This is the maximum variability in the sampling rate due to temperature over the range of 7.2–37.2°C (22.2±15°C or 72±27°F). When the atmospheric pressure is unknown, it will be determined from the estimated elevation of the sampling site. A value of 3% shall be used for SPr to account for pressure fluctuations due to weather conditions. This is an estimate of the variability due to weather, based on a study that tracked atmospheric pressure variations over a year's time at SLTC.

$$SEE = \sqrt{(SEE_R)^2 + (SR)^2 + (ST)^2 + (SPr)^2}$$

The precision at the 95% confidence level is obtained by multiplying the SEE by 1.96 (the z statistic from the standard normal distribution at the 95% confidence level).

SEEs and Precisions of the Overall Procedure at the 95% Confidence Interval for Adsorbent Tubes

Adsorbent	TWA Samples		Ceiling Samples		Peak Samples	
	SEE	Precision	SEE	Precision	SEE	Precision
Charcoal	5.51%	±10.8%	5.20%	±10.2%	5.25%	±10.3%
Anasorb®747	5.15%	±10.1%	5.14%	±10.1%	5.36%	±10.5%

¶. Precision (analytical method)

The precisions of the analytical procedure are defined as the pooled relative standard deviations (RSD_P). Relative standard deviations were determined from six replicate injections of standards at 0.5, 0.75, 1, 1.5 and 2 times the TWA target concentrations. After assuring that the RSDs satisfy the Cochran test for homogeneity at the 95% confidence level, the RSD_Ps were calculated to be 0.76%, 0.94% and 0.76% for the adsorbent tubes, 3M 3520 OVMs and SKC 575-002 samplers respectively.

Instrument Response for Adsorbent Tubes					
× target concn (µg/sample)	0.5×	0.75×	1.0×	1.5×	2.0×
peak areas (µV·s)	4541.2	6920.0	9082.5	13840	18165
	672250	1030700	1388800	2034600	2718600
	679710	1024500	1382300	2055000	2737100
	676130	1033400	1358700	2092000	2744500
	686650	1035000	1375800	2060800	2729200
	667840	1029400	1372900	2093000	2734000
	676290	1033100	1374800	2069600	2744000
mean	676479	1031017	1375550	2067500	2734567
SD	6427.5	3770.1	10126.2	22532.0	9778.9
RSD (%)	0.950	0.366	0.736	1.090	0.358

The Cochran test for homogeneity:

$$g = \frac{\text{largest RSD}^2}{\text{RSD}_{0.5x}^2 + \text{RSD}_{0.75x}^2 + \text{RSD}_{1x}^2 + \text{RSD}_{1.5x}^2 + \text{RSD}_{2x}^2}$$

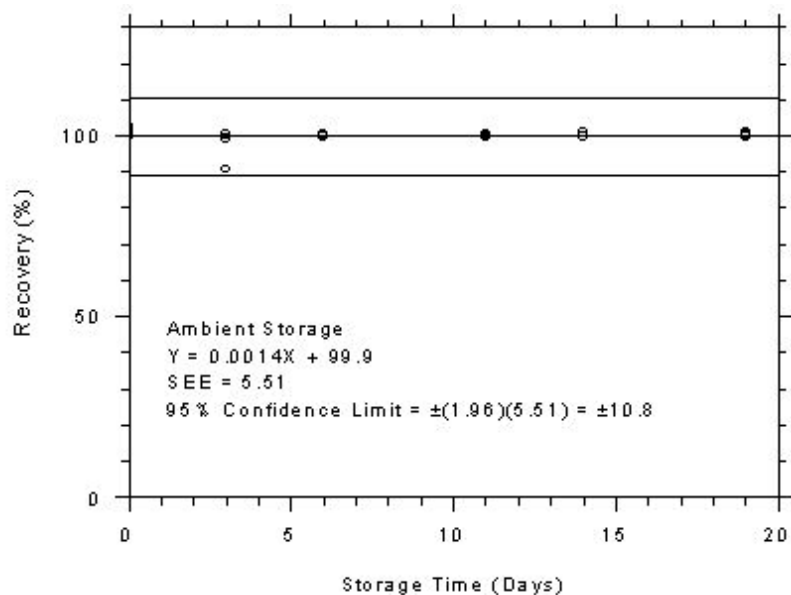
The g statistics are 0.410, 0.337 and 0.328 for the adsorbent tubes, 3M 3520 OVMs and SKC 575-002 samplers respectively. The critical value of the g statistic at the 95% confidence level for five variances, each associated with six observations, is 0.5065. Because the g statistics do not exceed this value, the RSDs can be considered equal and they can be pooled (RSD_P) to give an estimated RSD for the concentration range studied.

$$\text{RSD}_P = \sqrt{\frac{5(\text{RSD}_{0.5x}^2 + \text{RSD}_{0.75x}^2 + \text{RSD}_{1x}^2 + \text{RSD}_{1.5x}^2 + \text{RSD}_{2x}^2)}{5+5+5+5+5}}$$

사. Storage tests

Adsorbent tube storage samples were prepared by sampling at 50 mL/min from controlled test atmospheres that were at approximately 80% RH and at room temperatures ranging from 22–26°C and atmospheric pressures from 649–656 mmHg. Storage samples for 3M 3520 OVMs and SKC samplers were prepared by exposing them to the same atmospheres as for the adsorbent tubes. The flow of the atmospheres through the exposure chamber provided for face velocities on the diffusive samplers of approximately 0.4 m/s. TWA samples were prepared by sampling from 200-ppm atmospheres for 240 minutes, ceiling samples from 300-ppm atmospheres for 10 minutes and peak samples from 500-ppm atmospheres for 1 minute. Six samples for each set were analyzed immediately after generation, fifteen were stored in a refrigerator at 0°C and fifteen were stored in a closed drawer at ambient temperatures of 20–25°C. Only ambient storage tests were done for the ceiling and peak samples and only adsorbent tube samples were generated for the peak level. At approximately three-day intervals, three samples were selected from each of the storage sets and analyzed.

TWA Storage Tests for Charcoal Tubes						
time (days)	refrigerated storage recovery (%)			ambient storage recovery (%)		
0	100.6	102.0	99.9	100.6	102.0	99.9
0	102.8	100.4	101.3	102.8	100.4	101.3
3	99.7	99.1	101.4	100.2	90.7	99.2
6	101.7	101.3	101.3	100.0	99.7	100.2
11	99.8	100.4	99.4	99.5	100.1	100.1
14	99.4	101.4	99.9	99.6	99.8	100.6
19	98.1	99.5	100.8	100.3	99.8	100.8



Charcoal tubes ambient storage test, 240-minute samples at 200 ppm.

아. 중금속 분석의 검출한계, 정량한계(ICP)

1) Analytical Procedure

If necessary, determine detection limits using the manufacturer's software (if available). These limits normally do not significantly change during short time spans. A general rule is to recalculate detection limits

when an integral component (nebulizer, torch, mass flow controller, etc.) of the ICP has been replaced or adjusted. A typical calculation of detection limit (DL) is shown:

$$DL = (K \times SDI \times C)(I - I_0) \times S$$

where:

S : Solution volume in mL

K : Degree of confidence (sigma value)*

SDI : Standard deviation of reagent blank intensity (I_0)

C : Concentration of the calibration standard in $\mu\text{g/mL}$

I : Total intensity of standard containing concentration C

I_0 : Background intensity (reagent blank)

* In most cases, K=2 or 3 for qualitative and K=10 for quantitative determinations.

Reporting limits may be equal to or larger than the calculated detection limits. Reporting limits should be verified annually for each matrix.

2) Detection Limit of the overall procedure (DLOP) and reliable quantitation limit (RQL).

The DLOP is measured as mass per sample. Seven Ghost wipes were spiked with descending increments of analyte. These spiked Ghost Wipes, and a sample blank, were analyzed and the data obtained used to calculate the required parameters (standards error of estimate and the slope) for the calculation of the DLOP.

The RQL is considered the lower limit for precise quantitative measurements. It is determined from the regression line parameters obtained for the calculation of the DLOP, providing 75% to 125% of the analyte is recovered.

		$\mu\text{g/sample}$	Intensity		
		0	0.4		
		0.031	4.5		
		0.062	4.6		
		0.12	9		
		0.25	15.8		
		0.38	24.6		
		0.5	32.5		
		0.63	43		
회귀분석 통계량					
다중 상관계수	0.9976				
결정 계수	0.9952		3표준편차	3.41	
조정된 결정 계수	0.9944		DLOP	0.05	
표준 오차	1.1359		RQL	0.18	
관측수	8				
분산 분석					
	자유도	제곱합	제곱 평균	F 비	유의한 F
회귀	1	1616.958	1616.958	1253.135	3.39E-08
잔차	6	7.741981	1.29033		
계	7	1624.7			
	계수	표준 오차	t 통계량	P-값	하위 95%
Y 절편	0.85	0.603653	1.402044	0.21046	-0.63074
X 1	64.69	1.82736	35.39965	3.39E-08	60.21651

자. 중금속 분석의 검출한계 및 감도(AAS)

1) Qualitative detection limit

The concentration ($\mu\text{g/mL}$) of an element which would yield an absorbance (ABS) equal to twice the standard deviation of a series of measurements of an aqueous solution containing the element. The signal obtained from the aqueous solution must be distinctly greater than the baseline.

2) Analytical detection limit

The lowest concentration ($\mu\text{g/mL}$) of an element that can be reliably quantitated. This limit is the largest value obtained from any of the three calculations:

- a) Three times the smallest possible non-zero instrument reading,
- b) Two times the average baseline variation, or
- c) The lowest standard used to construct a concentration-response curve. One-tenth the concentration of this standard is considered to be the detection limit if:

The average reading for this standard is within 20% of its linear response. The linearity is determined by the other standards used to construct the least-squares curve fit.

If the lowest standard ABS reading is more than 20% in error, then an algorithm is used and the concentration value is increased in 10% increments until a concentration is achieved that would display less than 20% error or until the lowest standard concentration is reached.

3) Sensitivity

The concentration ($\mu\text{g/mL}$) of an element in aqueous solution which will produce an ABS of 0.0044 (8.6).

4) Linear Range

The working range of a specific analyte. The range is considered linear if doubling the concentration of a standard results in at least a 75% increase in ABS.

2. 영국 HSE의 검출한계 및 분석의 신뢰성 평가

MIDHS

*Methods for the Determination of
Hazardous Substances*
Health and Safety Laboratory



6/3

**Lead and inorganic
compounds of lead in air**

Laboratory method using flame or
electrothermal atomic absorption
spectrometry

March 1998

가. 검출한계

METHOD PERFORMANCE

Detection limits

19 The qualitative and quantitative detection limits for lead, defined as three times and ten times the standard deviation of a blank determination, have been determined¹⁰ to be $0.025 \mu\text{g ml}^{-1}$ and $0.084 \mu\text{g ml}^{-1}$ for flame atomic absorption spectrometry; and 0.30 ng ml^{-1} and 1.0 ng ml^{-1} for electrothermal atomic absorption spectrometry. For an air sample volume of 30 litres and a sample solution volume of 10 ml this corresponds to lead-in-air concentrations of $8.4 \mu\text{g m}^{-3}$ and $28 \mu\text{g m}^{-3}$ for flame atomic absorption spectrometry; and $0.10 \mu\text{g m}^{-3}$ and $0.34 \mu\text{g m}^{-3}$ for electrothermal atomic absorption spectrometry.

나. 분석의 신뢰성 평가

Overall uncertainty

30 The overall uncertainty for a measuring procedure is defined in BS EN 482¹¹ as 'the quantity used to characterise as a whole the uncertainty of the result given by a measuring procedure', and is expressed in percentage terms, by a combination of bias and precision according to the following equation:

$$OU = \frac{|\bar{X} - x_{ref}| + 2\sigma_{(n-1)}}{x_{ref}} \times 100\% \quad \text{Equation 3}$$

where : OU is the overall uncertainty of the procedure;

\bar{X} is the mean value of results of n repeated measurements;

x_{ref} is the true or accepted reference value; and

$\sigma_{(n-1)}$ is the standard deviation of n repeated measurements.

31 Equation 3 can be rewritten as:

$$OU = [|\text{bias}| + (2 \times RSD)] \times 100\% \quad \text{Equation 4}$$

where: bias is the difference between the mean measured concentration and the true or reference concentration, divided by the true or reference concentration, ie $\frac{(\bar{X} - x_{ref})}{x_{ref}}$; and

RSD is the relative standard deviation of n repeated measurements defined as $\frac{\sigma_{(n-1)}}{x_{ref}}$

3. NIOSH의 검출한계, 정량한계 평가

가. 검출한계, 정량한계 개념

Date Issued: January 24, 1984
Date Revised: July 18, 1994

SOP 018

Limits of Detection and Quantitation

- I. **Introduction** - This SOP addresses two measures of analytical capability for individual samples. Limit Detection (LOD) is a decision point used to decide whether to report a significant analyte signal from sample. Limit of Quantitation (LOQ) is the smallest amount of analyte which can be measured with precision. These parameters relate to the low-concentration end of the analytical working range and do not provide information about accuracy, precision, or sensitivity at higher concentrations.

[Note: This SOP applies to DPSE's routine chemical analyses on individual samples (excluding elemental analyses by ICP-AES and fiber counting by microscopy). Interpretation of results may be different from replicated factorial experiments; see a statistician for assistance in those cases.]

II. Definitions^[1]

- A. **Limit of Detection (LOD)** is the mass of analyte which gives a mean signal $3\sigma_b$ above the mean blank signal where σ_b is the standard deviation of the blank signal.
- B. **Limit of Quantitation (LOQ)** is the lowest mass that can be reported with acceptable precision. LOQ is the larger of:
- (a) the mass corresponding to the mean blank signal + $10\sigma_b$ (i.e., $\pm 30\%$ uncertainty), or
 - (b) the mass above which recovery is $\geq 75\%$.

나. 검출한계, 정량한계 평가 사례

VI. EXAMPLE - Pentamidine isethionate (Seq. 7292)

A. Analyze Low Standards [Steps A.3 and A.4, p.2]

pentamidine isethionate, ng/sample	response, peak area
0.153	214.8
0.306	739.3
0.615	1575
1.23	3790
2.46	6921
4.92	11526
9.84	21712
19.7	40033
39.4	82491

Notes: (Low standards in **bold**)

- Expected LOD = 1 ng.
- No response from field blanks
- Responses are for standard solutions, which were shown to give the same s_y as spiked filters.
- A separate recovery study was performed, giving 75% recovery at 50 ng/sample and 16% recovery at 1 ng/sample.

A linear regression using the above six low standards gives the equation:

$$Y = 280.9 + 2383.4 X \text{ (with } s_y = 603.8, \text{ slope RSD} = 0.062, \text{ and correlation coefficient} = 0.997).$$

B. Calculate LOD [Step A.5, p.2]

1. $LOD = 3s_y/\text{slope} = 3 \times 603.8/2383.4 = 0.76 \text{ ng per sample}$
2. At this level, the recovery is approximately 16%, so the recovery-corrected LOD is 0.76 divided by 0.16:

$$LOD = 5 \text{ ng/sample}$$

No Song-Fischbach correction is needed since the slope RSD is less than 0.09.


C. Calculate LOQ [Step A.6, p.2]

The LOQ is the larger of (a) $3.33 \times LOD$ and (b) the smallest mass with 75% recovery; in this case, (a) is larger and:

$$LOQ = 50 \text{ ng/sample}$$

III. 자료출처(참고할 수 있는 사이트)

주소(D)  <http://www.osha.gov/dts/sltc/methods/>



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Sampling and Analytical Methods

These procedures were designed and tested for internal use by OSHA personnel. Mention of any name or commercial product does not constitute endorsement by OSHA.

Index of Sampling and Analytical Methods

An [alphabetical list of chemicals](#) that have either a validated or partially validated OSHA method. Some chemicals may be listed by their common synonym. The index includes the method number, validation status, CAS number, analytical instrument and sampling device. A key for abbreviations is located at the end of the index.

Protocols for Methods Evaluation

Accessibility Assistance: Contact the OSHA Directorate of Science, Technology and Medicine, Salt Lake Technical Center at 801-233-4900 for assistance accessing OSHA PDF materials.

[Evaluation Guidelines for Air Sampling Methods Utilizing Chromatographic Analysis](#)

[Evaluation Guidelines for Air Sampling Methods Utilizing Spectroscopic Analysis](#)

[Method Evaluation Guidelines for Surface Sampling Methods](#)

What's New from the Methods Development Team at the OSHA Salt Lake Technical Center

- [Aminopyridine](#) - Method PV2143
- [Arsenic, Cadmium, Cobalt, Copper, Lead, and Nickel](#) - Method 1006
- [Ethylene Oxide](#) - Method 1010
- [Formaldehyde \(Diffusive Samplers\)](#) - Method 1007
- [Hexavalent Chromium](#) - Method ID-215 (Version 2)
- [Hydrogen Sulfide](#) - Method 1008
- [n-Amyl Acetate, Isoamyl Acetate](#) - PV2142
- [Scopolamine Methyl Nitrate](#) - PV2144

Current Projects

- Butyl Acetates. New method that permits active and diffusive sampling of butyl acetates is nearly complete.
- Sulfur Dioxide. New Active Sampling Method.

HSE - PUBLICATIONS: Methods for the Determination of Hazardous Substances (MDHS) guidance - Microsoft Internet Explorer

주소(C) http://www.hse.gov.uk/pubns/mdhs/index.htm

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Methods for the Determination of Hazardous Substances (MDHS) guidance

The following guidance is also available in hard copy from [HSE Books](#).

- MDHS guidance 3-25/3
- MDHS guidance 27-48
- MDHS guidance 52/3-75
- MDHS guidance 76-100
- MDHS guidance 101-125

MDHS guidance 3-25/3

- MDHS3 Generation of test atmospheres of organic vapours by the syringe injection technique Portable apparatus for laboratory and field use [200kb]
- MDHS4 Generation of test atmospheres of organic vapours by the permeation tube method Apparatus for laboratory use [192kb]
- MDHS6/3 Lead and inorganic compounds of lead in air Laboratory method using flame or electrothermal atomic absorption spectrometry [108kb]
- MDHS10/2 Cadmium and inorganic compounds of cadmium in air Laboratory method using flame atomic absorption spectrometry or electrothermal atomic absorption spectrometry [56kb]
- MDHS12/2 Chromium and inorganic compounds of chromium in air Laboratory method using flame atomic absorption spectrometry [100kb]
- MDHS14/3 General methods for sampling and gravimetric analysis of respirable and inhalable dust [128kb]

주소(D) http://www.cdc.gov/niosh/docs/95-117/

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NIOSH Publication No. 95-117: **May 1995**

Guidelines for Air Sampling and Analytical Method Development and Evaluation

The Occupational Safety and Health Act of 1970 charged the National Institute for Occupational Safety and Health with the responsibility for the development and evaluation of sampling and analytical methods for workplace compliance determinations. The purpose of this guideline document is to refine the original protocol for sampling and analytical method development and evaluation research with additional experiments to more fully evaluate method performance.

Entire Document [95-117.pdf](#) (4.5MB; 112 pgs)

The free Adobe Acrobat Reader is needed to view these files.

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Related Resources:

[NIOSH Manual of Analytical Methods](#)

Guidelines for Air Sampling and Analytical Method Development and Evaluation