Calculating the limit of detection (LOD) for a specific analyte with a spectrometer

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PLANNING INITIAL CONCENTRATION RANGE SCREENING

	Research the LOD of the analyte of interest for the phenomenon and technique desired. E.g., LOD for analyte "X" is 0.400 M.
	Determine the upper limit (UL) by multiplying the LOD by 5. E.g., UL=LOD·5 → UL=0.400 M·5 → UL=2.00 M
	Determine the increments by subtracting the UL from the lower limit (LL) and dividing by 4. Increments=(UL-LL)/4 → Increments=((2.00-0.400) M)/4 → Increments=0.400 M
	(OPTIONAL) IF LOD of the analyte of interest is not found, use the range from 50% to 10% at 10% increments.
	Determine the volume of the available flasks and the amount per volume.
	Determine the final volume of your solution (V2), i.e., the flask that will be used for the solution.
	Find the initial concentration (C1) of the analyte of interest.
	Determine the desired concentration of your solution (C2).
	Determine the volume needed for analyte with concentration C1 to reach the desired concentration (V1) with a final volume of V2. $V_1=(C_2 \cdot V_2)/C_1$
	(OPTIONAL IF AN INTEGER V1 VALUE IS NEEDED) Determine the final concentration C2 for the analyte with an initial concentration C1 after adding a specific initial volume V1, close to the value obtained from step 6, and completing to a final volume of V2. $C_2=(C_1 \cdot V_1)/V_2$
	Repeat steps 3-7 for the other solutions.
	IF an integer value of V1 cannot be obtained for a solution with the initial C1 concentration of the analyte of interest, use the final concentration C2 of a solution as the initial concentration C1 to determine the volume.
	Solution used to create other solutions might be consumed, create more, or change V2 to a bigger value.
	Prepare solutions by adding the initial volume of analyte (V1) of C1 concentration into the volumetric flask with the final volume (V2) to obtain the final concentration C2.

SCREENING LOD CONCENTRATION RANGE FOR ANALYTE

	Acquire spectra for each concentration and blank.
	10 measurements per sample. Average the measurements.
	Determine the peak with intensity proportional to the analyte concentration.
	Limit spectral data to the frequencies of the chosen spectral band.
	Calculate the area of the chosen peak with the first frequency (ν_1) and intensity (I_1) as well as the second frequency (ν_2) and intensity (I_2) with the following equation. $A=(\nu_2-\nu_1)\cdot((I_1+I_2)/2)$
	Repeat step 4 until the area for each frequency is calculated (first frequency does not contain area).
	Add the area for each frequency to obtain the area of the peak (PA). $PA=\sum_{i=1}^{n}[A_i]$ → PA=0+1.195+1.455+1.693+1.838+1.810+1.545+1.255+1.095+0.954 → PA=12.841
	Repeat steps 4-6 for all samples.
	Plot peak area vs concentration.
	Calculate slope and intercept error by using the =LINEST function.
	Select the 'y' values, then the 'x' values, write "TRUE" twice, and click enter. A range of values will appear below (2 columns and 5 rows).
	Names will not appear.
	Add trendline by selecting the data points, right click, and choose "Add Trendline...".
	Write linear equation with R2 and errors. y=slope(±standard error m)+intercept(±standard error b) R^2
	Calculate standard deviation (s) for each concentration. =STDEV(Range of values)
	Add error bars by selecting the data points, selecting the "Chart Design" tab, "Add Chart Element" button, "Error Bars" list element, and "More Error Bars Options" option.
	Use standard deviation by going to "Custom" in Error Amount and "Specify Value".
	Select standard deviation values in both positive and negative error values.
	Calculate relative standard deviation (RSD) for the first row by dividing the average peak area with the standard deviation. $RSD=s/\bar{x}\times100\%$
	Repeat step 9 for the next peak.
	Repeat step 10 for row with peak values.
	Graph relative standard deviation (RSD) vs concentration.
	Determine the concentration range by analyzing the RSD values.
	Values above 10% can be discarded from the desired LOD range. E.g., the concentration range for this data set is 9.9 – 35%v/v.

CALCULATING LOD FOR SPECIFIC ANALYTE

	Calculate the increment concentrations for the range determined in the previous section (II).
	E.g., calculate the increments for the range 9.9 – 35.5%v/v for 5 samples. Increments=(UL-LL)/(n-1) → Increments=((35.5-9.9) %)/(5-1) → Increments=6.4 %
	Prepare solutions as shown in section I. V_1=(C_2·V_2)/C_1
	Acquire spectra for each sample.
	Repeat step 3 until 10 measurements are acquired for each concentration and 20 measurements for the blank.
	Calculate the area for the peak of interest for each concentration as in step II. A=(v_2-v_1)·((I_1+I_2)/2)
	Plot peak area vs concentration.
	Register the slope (m) for the calibration curve. y=0.0069(±0.0004)+0.0609(±0.0103) → m=0.0069
	Calculate the average for all 20 peak area measurements for the blank sample. S¯_bl=(∑_(i=1)^n[PA_i])/n
	Calculate the standard deviation for all 20 peak area measurements for the blank sample. s_bl=√((∑_(i=1)^n[(PA_i-(PA)¯)^2])/(n-1))
	Calculate the LOD with the average (S¯_bl), standard deviation (s_bl), and regression slope (m). LOD=((S¯_bl+3.3·s_bl)-S¯_bl)/m → LOD=((0.018+3.3·0.006)-0.018)/0.0069 → LOD=2.9%
	Calculate the LOQ with the average (S¯_bl), standard deviation (s_bl), and regression slope (m). LOQ=((S¯_bl+10·s_bl)-S¯_bl)/m → LOQ=((0.018+10·0.006)-0.018)/0.0069 → LOQ=8.7%