

California Environmental Protection Agency



CALIFORNIA
AIR RESOURCES BOARD

PROCEDURE FOR THE ANALYSIS OF AUTOMOTIVE EXHAUST FOR METHANOL AND ETHANOL

**Standard Operating Procedure No. MV-NMOG-101
Revision 2.3**

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**SOP No. 101 - PROCEDURE FOR THE ANALYSIS OF AUTOMOTIVE EXHAUST
FOR METHANOL AND ETHANOL**

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1 Introduction

- 1.1 This document describes a method for analyzing methanol and ethanol in automotive exhaust in the range of 1 to 1200 micrograms (μg) per 15 milliliter (mL) of solution. Other alcohols soluble in water may also be determined should peak identification and calibrations be performed.
- 1.2 This procedure is based on a method developed by the U.S. Environmental Protection Agency (EPA, Ref. 11.1) which involves sampling diluted engine exhaust through deionized water contained in glass impingers and analyzing this solution by gas chromatography. This method does not encompass sample collection procedures.
- 1.3 This SOP is based on Method 1001 of the California Non-methane Organic Gas Test Procedures Part C, *California Air Resources Board*, Amended March 22, 2012
- 1.4 High concentrations may be determined by quantitatively diluting the aqueous solution with deionized water.

2 Method Summary

- 2.1 Typically a pair of impingers, one primary one back up, is connected in series to be collected for each sample.
- 2.2 The samples are then brought to the laboratory and place in the refrigerator.
- 2.3 An aliquot of each sample is transferred to an autosampler vial. The sample is introduced into a gas chromatograph (GC) by the liquid autosampler. The alcohols in the sample are separated in a GC capillary column and subsequently detected and quantified by a flame ionization detector (FID).

3 Interferences and Limitations

- 3.1 An interferent is any component present in the sample with a retention time similar to that of any target alcohol described in this method. To reduce interference error, proof of chemical identity may require periodic confirmations using an alternate method or instrumentation, e.g., GC/MS.
- 3.2 The concentration of the alcohols in the range of interest is stable for up to six days as long as the samples are sealed and refrigerated at approximately 40 °F. Therefore, samples must be kept refrigerated and be analyzed within six days of sampling.

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4 Instrumentation and Apparatus

- 4.1 The analytical system is comprised of the following:
 - 4.1.1 Gas chromatograph (GC) (i.e. Varian 3800) equipped with DB-Wax Megabore column [30 meters, 0.53 millimeters (mm) internal diameter (ID), 1 micron (μ) film thickness] and flame ionization detector (FID). Other columns may be used, provided the alternate(s) can be demonstrated to be equivalent or better with respect to precision, accuracy, and resolution of all the target compounds.
 - 4.1.2 An autosampler (i.e., Varian model CP8400) capable of keeping the sample cool with a refrigerated, re-circulating water bath.
 - 4.1.3 Instrument control software with data processing capability (i.e., Varian Star version 6.0).

5 Reagents and Materials

- 5.1 Methanol, 99.9%, HPLC grade.
- 5.2 Ethanol, absolute, ACS reagent grade.
- 5.3 ASTM Type I deionized water, HPLC grade.
- 5.4 Stock solutions are prepared by diluting approximately 1 g (must be weighed with an analytical balance with readability of 0.1 mg) each of methanol and ethanol with high purity water to the mark of a 100.0 mL flask. The concentrations of these standards are calculated and expressed in $\mu\text{g/mL}$.
 - 5.4.1 Stock solutions must be prepared at least every six months.
 - 5.4.2 Calibration standards are prepared by successive dilutions of the stock solution(s). A typical calibration standard is 3.0 $\mu\text{g/mL}$, though higher concentration standards should be used for high concentration samples. Calibration standards must be replaced at least every week.
 - 5.4.3 A control standard is also prepared by successive dilutions of a stock solution different from that of the calibration standard. The concentration should be approximately that of the samples, typically 1.5 $\mu\text{g/mL}$. Control standards must be replaced at least every week.
 - 5.4.4 Standards used for linearity and LOD determinations (Section 9) are also prepared by successive dilutions of an appropriate level stock solution.
 - 5.4.5 All standards should be refrigerated at approximately 40 °F during storage.

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- 5.5 If other alcohols are found in the exhaust, standards containing these additional compounds are prepared, as above.
- 5.6 Gas requirements:
 - 5.6.1 Air shall be "Zero" grade (<1 ppmC total hydrocarbon contamination) or better.
 - 5.6.2 Nitrogen shall have a minimum purity of 99.998 percent.
 - 5.6.3 Helium shall have a minimum purity of 99.995 percent.
 - 5.6.4 Hydrogen shall have a minimum purity of 99.995 percent.

6 Safety

- 6.1 Methanol, ethanol, and other alcohols can be harmful or fatal if inhaled.
- 6.2 Prepare alcohol standards under a fume hood that has adequate ventilation.
- 6.3 For general laboratory safety procedures, consult the CAERB Chemical Hygiene Plan. Safety Data Sheets (SDSs) for each standard gas and chemical used are required to be kept in the laboratory.
- 6.4 Wear safety glasses and disposable gloves when handling liquid chemicals or fuels.
- 6.5 All compressed gas cylinders present hazards and should be handled appropriately. In addition to the potential hazard of all high-pressure gasses, hydrogen is extremely flammable.
- 6.6 Additional safety-related information can be found in CARB's Illness and Injury Prevention Plan (IIPP).
- 6.7 Staff must complete periodic safety education and/or training, as specified by branch policies.

7 Procedure

- 7.1 After sampling, the solution is either analyzed immediately or stored at approximately 40°F.
- 7.2 Prior to analysis, an aliquot of 1 to 2 mL of each sample is transferred to a 2-mL autosampler vial.
- 7.3 Place the samples in the autosampler and allow the vials to equilibrate in temperature (i.e. 11-15 °C).

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- 7.4 A 1.0 microliter (μL) aliquot of each unmodified sample is injected via autosampler into a gas chromatograph. The configurations below are provided as examples, but may differ between instruments.

Column	DB-wax, 30 m, 0.53 mm ID, 1.0 μ film thickness
Carrier gas	helium at 5 mL/minute (mL/min)
Make-up gas	nitrogen at 25 mL/min
Detector	FID, hydrogen at 30 mL/min, air at 300 mL/min; 200°C
Injector	on-column injection; 150°C
Autosampler temperature (if applicable)	11°C
Temperature	50°C (hold 8 min), 50°C to 110°C (15°C/min), 110°C (hold 3 min)

- 7.5 Samples containing compounds having concentrations above the documented range of instrument linearity must be diluted and reanalyzed.
- 7.6 The peak integrations are corrected as necessary in the data system. Any misplaced baseline segments are corrected in the reconstructed chromatogram.
- 7.7 The peak identifications provided by the computer are checked and corrected if necessary.
- 7.8 The above procedure may be modified for analysis of higher alcohols by increasing the final temperature of the running method and adjusting the temperature ramping to achieve good separation of the desired components.

8 Calculations

- 8.1 The concentration of each alcohol is can be determined by the single-point calibration.
- 8.2 The concentration of each alcohol is determined by comparing the sample peak area with that of an external standard:

$$\text{Concentration } (\mu\text{g/mL})_{\text{Sample}} = \text{PeakArea}_{\text{Sample}} * \text{ResponseFactor}$$

where the response factor (RF) is calculated during the calibration by:

$$RF = \frac{\text{Concentration}_{\text{Standard}} (\mu\text{g/mL})}{\text{Peak Area}_{\text{Standard}}}$$

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- 8.4 This concentration is then used to calculate the total amount of methanol and ethanol in each impinger:

$$Mass (\mu g) = Concentration (\mu g/mL) * Impinger Volume$$

Impinger volumes are typically 15 mL.

9 Quality Control

9.1 Blank Runs

- 9.1.1 A deionized water blank is run each analysis day to check the water used for sampling and the analytical system for contamination.
- 9.1.2 If the blank shows a peak greater than the limit of detection (LOD) in the region of interest, the blank analysis is repeated.
- 9.1.3 If the peak area is consistent, the blank value is subtracted from the samples.
- 9.1.4 If the peak area is not consistent, the source of the contamination must be investigated.
- 9.1.5 A trip blank is also analyzed for every emission test.
- 9.1.6 If the blank shows a peak greater than the limit of detection (LOD) in the region of interest, the blank value is subtracted from the samples.
- 9.2 Calibration Run: A single-point calibration is performed for each analysis day. Figure 1 illustrates typical chromatograms of a methanol/ethanol standard. The sample load frequently requires continuous instrument operation into the next calendar day. In these instances, the calibration factor of the previous calendar day is used for all of the samples in the same set.

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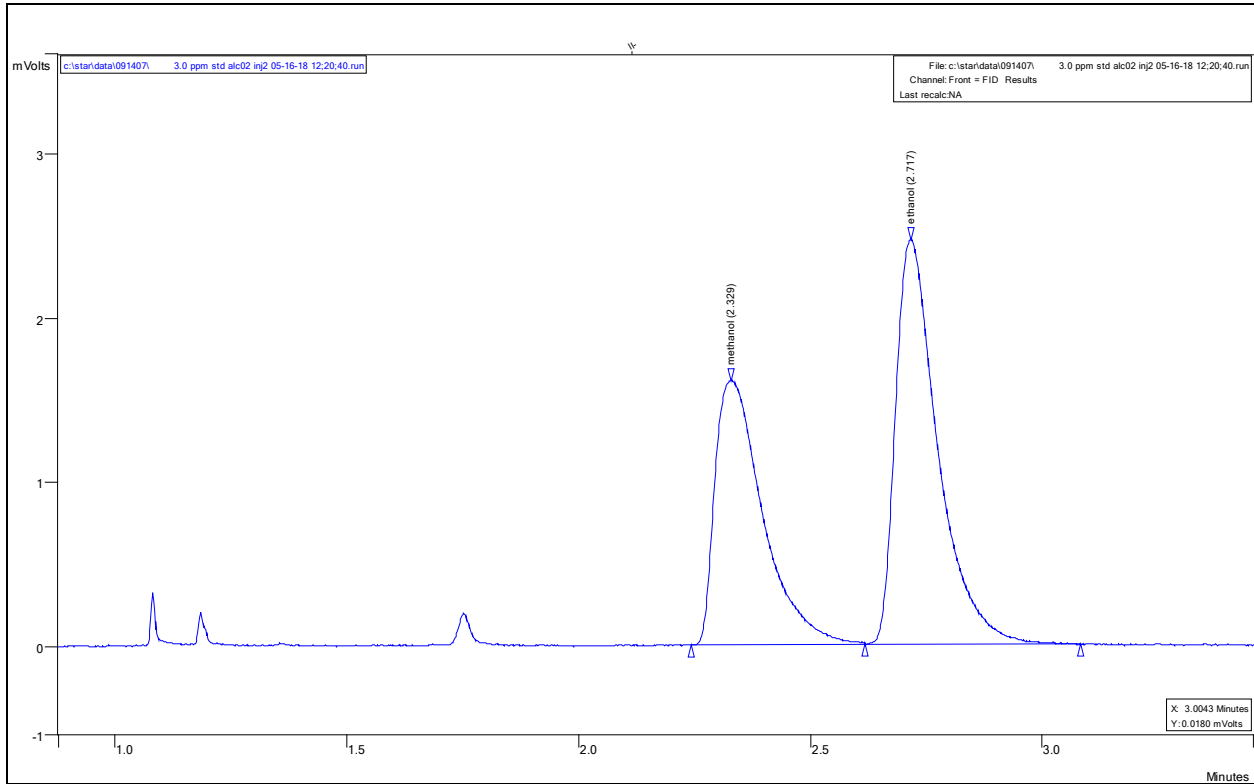


Figure 1. Typical Elution Profile of a 3 µg/mL Calibration Standard

9.2.1 A response factor for each target alcohol is generated daily by entering the concentration and measured peak area for each alcohol. Figure 2 demonstrates a typical calibration standard chart.

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No	Injection Date	Sequence	METHANOL 3						ETHANOL 3						Overall Status
			Calibration Factor	Area Count	Standard $\mu\text{g/mL}$	Calibration Mean	Calibration σ	Status	Calibration Factor	Area Count	Standard $\mu\text{g/mL}$	Calibration Mean	Calibration σ	Status	
1	8/18/2015 0:00	1.seq	3700	11404	3.082				5526	17247	3.121				
2	8/20/2015 0:00	1.seq	3855	11881	3.082				5591	17449	3.121				
3	8/21/2015 0:00	1.seq	3766	11500	3.054				5395	16902	3.133				
4	8/26/2015 0:00	1.seq	4081	12607	3.089				5889	18420	3.128				
5	8/27/2015 0:00	1.seq	3026	9367	3.095				5328	16698	3.134				
6	8/28/2015 0:00	1.seq	3475	10765	3.098				5113	16041	3.137				
7	8/31/2015 0:00	1.seq	4040	12596	3.118				5910	18663	3.158				
8	9/1/2015 0:00	1.seq	3910	12180	3.115				5592	17636	3.154				
9	9/2/2015 0:00	1.seq	3797	11971	3.153				5510	17587	3.192				
10	9/4/2015 0:00	1.seq	3891	12241	3.146				5752	18327	3.186				
11	9/9/2015 0:00	1.seq	3915	12136	3.100				5669	17796	3.139				
12	9/16/2015 0:00	1.seq	3987	12233	3.068				5633	17501	3.107				
13	9/17/2015 0:00	1.seq	3869	11902	3.076				5479	17067	3.115				
14	9/29/2015 0:00	1.seq	3826	11741	3.069				5573	17320	3.108				
15	10/15/2015 0:00	1.seq	3849	11867	3.083				5637	17592	3.121				
16	10/19/2015 0:00	1.seq	4092	12631	3.087				5828	18219	3.126				
17	10/26/2015 0:00	1.seq	2991	9305	3.111				4427	13946	3.150				
18	10/27/2015 0:00	1.seq	3912	12138	3.103				5693	17887	3.142				
19	11/6/2015 0:00	1.seq	5345	16629	3.111				5472	16412	2.999				
20	11/9/2015 0:00	1.seq	3806	10658	2.800				5505	16918	3.073				
1	1/20/2016 0:00	1.seq	4077	12460	3.056	3857	459	pass	5729	17903	3.125	5526	320	pass	
2	1/26/2016 0:00	1.seq	3907	11991	3.069	3867	450	pass	5499	17255	3.138	5536	315	pass	
3	2/2/2016 0:00	1.seq	3874	11917	3.076	3867	450	pass	5471	17207	3.145	5536	315	pass	
4	2/5/2016 0:00	1.seq	4012	12242	3.051	3869	439	pass	5707	17807	3.120	5534	307	pass	
5	2/8/2016 0:00	1.seq	4058	12384	3.052	3869	429	pass	5708	17810	3.120	5531	301	pass	
6	2/12/2016 0:00	1.seq	3942	12043	3.055	3875	421	pass	5533	17284	3.124	5539	296	pass	
7	3/18/2016 0:00	1.seq	4152	12709	3.061	3882	414	pass	5839	18275	3.130	5545	292	pass	

Figure 2. Calibration Standard Control Chart

- 9.2.2 A running mean response factor and standard deviation for each alcohol is calculated and used for the criteria of a calibration standard check.
- 9.2.3 The instrument or the conditions of the analysis need to be investigated if the measured response factor is outside 3 standard deviations or 10 % from current mean response factor, whichever is greater.
- 9.2.4 If major problems are discovered or the cause of the failure is unknown, samples should not be analyzed by the GC.
- 9.2.5 Instrument maintenance and repairs can affect the instrument response. If the response changes sufficiently that the subsequent calibration fails the control limits, a new calibration chart should be started.
- 9.2.6 In the event that twenty successful calibration analyses have not been performed before the need arises for running samples, the calibration is considered valid if the control standard run (Section 9.3) passes.
- 9.3 **Control Standard Run:** The quality control standard is analyzed at the beginning of each set of samples, repeated approximately every ten samples, and after the last sample.
- 9.3.1 Obtain at least 20 control standard results to create a control chart and establish control limits. Figure 3 demonstrates a typical set of control standard QC charts.

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No	Injection Date	METHANOL						ETHANOL					
		Normalized	Measured	Theoretical Value	Average	σ	Status	Normalized	Measured	Theoretical Value	Average	σ	Status
1	9/29/2015 0:00	0.895	1.415	1.581				0.945	1.504	1.592			
2	9/29/2015 0:00	1.011	1.598	1.581				0.997	1.587	1.592			
3	9/29/2015 0:00	0.951	1.503	1.581				0.980	1.560	1.592			
4	10/15/2015 0:00	0.979	1.475	1.507				1.008	1.538	1.526			
5	10/15/2015 0:00	0.983	1.481	1.507				1.018	1.554	1.526			
6	10/15/2015 0:00	1.032	1.555	1.507				0.983	1.530	1.556			
7	10/19/2015 0:00	0.858	1.318	1.537				0.927	1.442	1.556			
8	10/19/2015 0:00	0.848	1.303	1.537				0.947	1.474	1.556			
9	10/19/2015 0:00	0.992	1.524	1.537				1.000	1.556	1.556			
10	10/26/2615 0:00	1.012	1.543	1.524				1.026	1.583	1.543			
11	10/26/2615 0:00	1.001	1.525	1.524				1.038	1.601	1.543			
12	10/26/2615 0:00	0.977	1.489	1.524				1.005	1.550	1.543			
13	10/27/2615 0:00	0.970	1.507	1.554				0.984	1.548	1.573			
14	10/27/2615 0:00	0.840	1.306	1.554				0.955	1.502	1.573			
15	10/27/2615 0:00	1.006	1.564	1.554				1.009	1.587	1.573			
16	11/6/2015 0:00	0.999	1.563	1.565				1.021	1.541	1.509			
17	11/6/2015 0:00	0.919	1.438	1.565				1.004	1.515	1.509			
18	11/6/2015 0:00	1.016	1.590	1.565				1.043	1.574	1.509			
19	11/9/2015 0:00	0.864	1.355	1.568				0.991	1.498	1.512			
20	11/9/2015 0:00	0.846	1.327	1.568				0.987	1.492	1.512			
1	1/20/2016 0:00	1.038	1.588	1.530	0.950	0.067	pass	1.007	1.575	1.564	0.993	0.031	pass
2	1/20/2016 0:00	1.016	1.554	1.530	0.954	0.068	pass	1.007	1.561	1.564	0.994	0.031	pass
3	1/20/2016 0:00	1.008	1.543	1.530	0.957	0.067	pass	0.998	1.576	1.564	0.995	0.030	pass
4	1/26/2016 0:00	1.010	1.552	1.537	0.959	0.067	pass	1.008	1.583	1.571	0.995	0.029	pass
5	1/26/2016 0:00	1.003	1.542	1.537	0.961	0.066	pass	1.008	1.572	1.571	0.995	0.029	pass
6	1/26/2016 0:00	1.009	1.551	1.537	0.966	0.065	pass	1.001	1.589	1.571	0.996	0.028	pass
7	2/2/2016 0:00	1.042	1.616	1.554	0.965	0.065	pass	1.011	1.620	1.588	0.996	0.028	pass
8	2/2/2016 0:00	1.021	1.587	1.554	0.968	0.065	pass	1.004	1.594	1.588	0.997	0.027	pass
9	2/2/2016 0:00	1.043	1.621	1.554	0.969	0.065	pass	1.020	1.619	1.588	0.997	0.027	pass
10	2/5/2016 0:00	1.016	1.574	1.549	0.972	0.065	pass	1.003	1.588	1.584	0.998	0.027	pass
11	2/5/2016 0:00	0.994	1.539	1.549	0.973	0.064	pass	0.998	1.581	1.584	0.998	0.026	pass
12	2/5/2016 0:00	0.986	1.527	1.549	0.974	0.063	pass	1.001	1.586	1.584	0.998	0.026	pass
13	2/8/2016 0:00	1.010	1.558	1.543	0.974	0.062	pass	1.006	1.588	1.578	0.998	0.025	pass
14	2/8/2016 0:00	0.990	1.528	1.543	0.976	0.062	pass	0.972	1.534	1.578	0.998	0.025	pass
15	2/8/2016 0:00	1.003	1.557	1.543	0.976	0.061	pass	1.001	1.579	1.578	0.997	0.025	pass
16	2/12/2016 0:00	1.003	1.533	1.529	0.977	0.060	pass	0.968	1.514	1.564	0.997	0.025	pass
17	2/12/2016 0:00	1.005	1.537	1.529	0.977	0.059	pass	0.987	1.544	1.564	0.997	0.025	pass
18	2/12/2016 0:00	0.969	1.481	1.529	0.978	0.059	pass	0.976	1.527	1.564	0.996	0.025	pass
19	3/18/2016 0:00	0.970	1.496	1.542	0.978	0.058	pass	0.959	1.513	1.577	0.996	0.024	pass
20	3/18/2016 0:00	0.970	1.495	1.542	0.978	0.057	pass	0.982	1.549	1.577	0.995	0.025	pass
21	3/18/2016 0:00	0.958	1.478	1.542	0.978	0.056	pass	0.977	1.540	1.577	0.995	0.025	pass

Figure 3. Quality Control Chart

9.3.2 Calculate the control standard mean from the ratio of the measured value to the theoretical value for each target alcohol.

9.3.3 Calculate the control standard deviation from the ratio of the measured value to the theoretical value for each target alcohol.

9.3.4 Establish upper and lower warning limits at either two standard deviations or 5 percent, whichever is greater, above and below the mean ratio. A measured ratio outside of this limit is considered a QC warning. When warnings occur on

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two consecutive analyses, the second analysis is considered a QC failure.

- 9.3.5 Establish an upper and lower QC control limits at either three standard deviations or 5 percent, whichever is greater, above and below the mean ratio. A measured ratio outside this limit is considered a QC failure.
- 9.3.6 In the event that twenty successful control standard analyses have not been performed before the need for running samples, an alternative QC criterion will be used. In this case, a measured concentration greater than 10% from the theoretical concentration is considered a QC failure.
- 9.3.7 A QC failure requires that the instrument and the conditions of analysis be investigated before running samples. If major problems are discovered or the cause of the failure is unknown, samples should not be analyzed by the GC. Any sample analyses performed are considered invalid.
- 9.4 Replicate Run: A replicate analysis performed on one of approximately every 10 samples, or at least once per analysis day. The relative percent difference (RPD) in concentration between the pair of analyses is calculated for each of the target alcohols and compared to an allowable limit. A sample replicate chart is shown in Figure 4.

Run No.	Date	TEST ID	Sample ID	METHANOL					ETHANOL				
				Run 1	Run 2	% RSD	Max RSD	Status	Run 1	Run 2	% RSD	Max RSD	Status
1	1/20/2016	217-H686-1	S1	0.190	0.177	7.1	15.00	PASS	0.465	0.455	2.2	15.00	PASS
2	1/26/2016	25EC1B-4	S1	0.199	0.205	3.0	15.00	PASS	2.025	2.001	1.2	15.00	PASS
3	2/5/2016	245D72E-1	S1	0.094	0.088	6.6	15.00	PASS	12.302	12.120	1.5	15.00	PASS
4	2/8/2016	24EC1B-4	S1	<LOD	<LOD	NA	NA	PASS	0.736	0.767	4.1	15.00	PASS
5	2/12/2016	25D72E-1	S1	0.140	0.135	3.6	15.00	PASS	8.319	8.215	1.3	15.00	PASS
6	3/18/2016	26D72E-1	S1	0.102	0.092	10.3	15.00	PASS	13.040	12.898	1.1	15	PASS

Figure 4. Replicate control chart

- 9.4.1 The RPD is calculated as follows:

$$RPD = \frac{|Sample\ Conc. - Replicate\ Conc. |}{Average\ Conc.\ of\ Both\ Analyses} \times 100$$

- 9.4.2 A limit on the allowable RPD is established based on the average concentration of the replicate runs, as shown in the following table:

Average Measurement for Replicate Runs	Allowable RPD (%)
1 to 10 times LOD	100
10 to 20 times LOD	30
20 to 50 times LOD	20
Greater than 50 times LOD	15

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- 9.4.3 If the measured RPD of any of the target compounds is greater than the allowable limit, the sample should be analyzed again. If reanalysis is not feasible or if the RPD criteria are still not met on reanalysis, all of the sample results for that analysis day from the instrument are considered invalid.
- 9.5 Linearity Determination: A multipoint calibration of each of the target alcohols is performed to confirm instrument linearity. This is done for new instruments, after making instrument modifications which can affect linearity, and at least once per year.
- 9.5.1 The multipoint calibration consists of analyses of at least five concentration levels of standards distributed over the range of expected sample concentration. Each concentration level is measured at least twice. A linear regression analysis based on the 'least squares' method is performed using concentration as the independent variable and peak area as the dependent variable to determine a correlation coefficient (r). The r must be greater than 0.995 for the method to be considered sufficiently linear to ensure the validity of using a single-point calibration for daily analysis.
- 9.6 LOD Determination: A limit of detection (LOD) determination for each of the target alcohols is performed for new instruments, after making modifications which can affect the sensitivity of an instrument, and at least once per year. Figure 5 shows a typical LOD determination.

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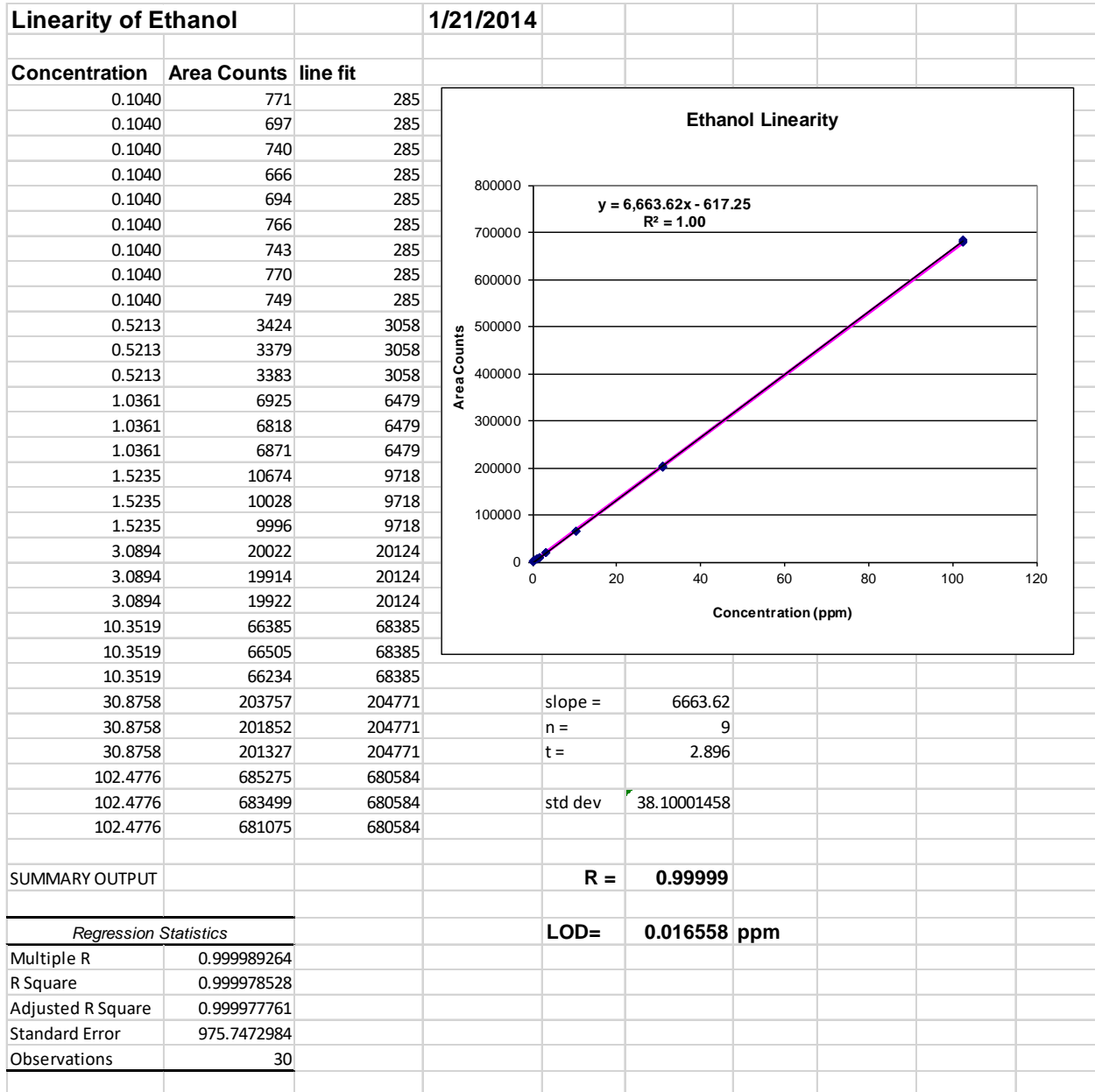


Figure 5. Linearity and LOD Determination for Ethanol

- 9.6.1 LOD determination consists of analyses of at least four “low” concentration levels standards, each above the LOD, with at least five replicate determinations of the lowest concentration standard.
- 9.6.2 The LOD determination can be performed concurrently with the linearity determination (Section 8.5) if at least five replicate measurements of the lowest concentration level standard are performed.
- 9.6.3 The concentration of the lowest standard must be greater than the calculated laboratory LOD, and not more than five times the estimated LOD.

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- 9.6.4 A linear regression analysis is performed on this data set to identify slopes, m_i , for each of the i th target alcohols.
- 9.6.5 For each of the i th target compounds, the standard deviations, s_i , in units of peak area are determined using the five (or more) replicate measurements of the lowest concentration standard. These are then converted to units of concentration using the slopes determined in Section 8.6.1.
- 9.6.6 Linearity and LOD determinations can be performed in the same set of experiments.

$$s_i^{conc} = \frac{s_i^{area}}{m_i}$$

- 9.6.6 The LOD for each of the i th target compounds can now be calculated using the following equation:

$$LOD_i = t * s_i^{conc}$$

where t is the Student's t value associated with a 98% confidence interval.

- 9.6.7 The Student's t value is dependent upon the degrees of freedom associated with the analysis. The degrees of freedom of the analysis is equal to the number of replicate measurements, n , of the lowest concentration standard minus one. An abbreviated table of values of t associated with a 98% confidence interval is shown below (Ref. 10.2):

Degrees of Freedom (n-1)	t-value
4	3.7
5	3.4
6	3.1
7	3.0

- 9.6.8 The maximum allowable LOD for each compound is 0.10 $\mu\text{g/mL}$. The calculated laboratory LOD must be equal to or lower than the maximum allowable LOD for sample analyses to be considered valid.
- 9.6.9 For sample analysis, all peaks identified as target compounds that are equal to or greater than the maximum allowable LOD must be reported. If the calculated laboratory LOD is less than the maximum allowable LOD, SLB may set its reporting limit at the maximum allowable LOD, the calculated laboratory LOD, or any level in between.

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9.6.10 For the purpose of calculating the total mass of all species, the concentrations of all compounds below the LOD are considered to be zero.

10 References

- 10.1 "EPA Method Characterization of Exhaust Emissions from Methanol and Gasoline Fueled Automobiles", EPA 460/3-82-004, U.S. Environmental Protection Agency.
- 10.2 Harris, Daniel C., "Quantitative Chemical Analysis", *W.H. Freeman & Co.*, 4th ed., 1995.
- 10.3 "EPA Method 8260D (SW-846): Volatile Organic Compounds by Gas Chromatography-Mass Spectrometry (GC/MS)", Revision 4, U.S. Environmental Protection Agency, February 2017.
- 10.4 "EPA Method 8015D (SW-846): Nonhalogenated Organics Using GC/FID", Revision 4, U.S. Environmental Protection Agency, February 2003.

11 Revision History

SOP No. MLD 101, Revision 2.2, January 2005

SOP No. MLD 101, Revision 2.1, April 2001

SOP No. MLD 101, Revision 2.0, September 1, 1996

SOP No. MLD 101, Revision 1.1, July 1993

SOP No. MLD 101, Revision 1.0, November 1, 1989