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Verification of LC-MS Method Suitability for Application to OPCW Proficiency Testing

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ABSTRACT

Defence Science and Technology (DST) Group is working towards becoming a designated laboratory for the Organisation for the Prohibition of Chemical Weapons (OPCW), and thus participates in its proficiency testing program. DST has typically relied on a combination of data from Gas Chromatography-Mass Spectrometry with Electron Impact (GC-MS[EI]) and Gas Chromatography-Mass Spectrometry with Chemical Ionisation (GC-MS[CI]) for reporting purposes, however it has recently implemented a Liquid Chromatography-Mass Spectrometry (LC-MS) method as a third technique to provide additional information where required. Given that samples received as part of the program may be spiked with compounds at concentrations as low as 1 ppm, this document reports the work undertaken in ascertaining that the limit of detection (LOD) of the LC-MS method is below this threshold, and that the method is overall fit for application in OPCW proficiency tests. The results of the work demonstrate that for all compounds assessed, the LOD is below 1 ppm and the method is acceptably repeatable so that it may be used for reporting in OPCW proficiency tests.

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Executive Summary

Defence Science and Technology (DST) Group is working towards achieving designated laboratory status with the Organisation for the Prohibition of Chemical Weapons (OPCW). Such laboratories are those which may be used by the OPCW to perform offsite analysis to assist in its investigations pertaining to verification of destruction or alleged use of chemical weapons agents (CWAs). Accordingly, DST regularly participates in the OPCW proficiency testing program to satisfy the OPCW's condition of at least annual participation in the program, and an A-grade majority maintained across every three tests in which the laboratory has participated.

During a proficiency test, participating laboratories have 15 calendar days in which to analyse the samples and report results. Results reported must have supporting evidence provided by at least two analytical techniques. DST has typically relied on a combination of Gas Chromatography-Mass Spectrometry with Electron Impact (GC-MS[EI]) and Gas Chromatography-Mass Spectrometry with Chemical Ionisation (GC-MS[CI]) data, however it has recently implemented a Liquid Chromatography-Mass Spectrometry (LC-MS) method as a third technique to provide additional information where required. Given that samples received as part of the program may be spiked with compounds at concentrations as low as 1 ppm, this document reports the work undertaken in the verification of the limit of detection (LOD) of the LC-MS method as being below this level, and that the method is overall fit for application in OPCW proficiency tests.

Herein, solutions of nine hydrolysis products of various (eight) CWAs were prepared at six concentrations in the range 0.25 to 10 ppm, and analysed using the aforementioned method. Results showed that eight of the nine compounds demonstrate a high degree of linearity over the concentration range 0.25 to 10 ppm, and all compounds had an LOD below the 1 ppm threshold, with acceptable repeatability, rendering the LC-MS method fit for participation in OPCW proficiency tests.

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Abbreviations

CAAF	Chemical Agents Analysis Facility
CMPA	Cyclohexyl Methylphosphonic Acid
CWA	Chemical Warfare Agent
DST Group	Defence Science and Technology Group
EDMAP	O-Ethyl-N,N-dimethylamido Phosphoric Acid
EIC	Extracted Ion Chromatogram
EMPA	Ethyl Methylphosphonic Acid
EPA	Ethylphosphonic Acid
GA	Tabun
GB	Sarin
GC-MS(CI)	Gas Chromatography-Mass Spectrometry with Chemical Ionisation
GC-MS(EI)	Gas Chromatography-Mass Spectrometry with Electron Impact
GD	Soman
GF	Cyclosarin
HD	Sulfur Mustard
HN2	Nitrogen Mustard
IMPA	Isopropyl Methylphosphonic Acid
ISO	International Organisation for Standardisation
LC	Liquid Chromatograph(y)
LC-MS	Liquid Chromatography-Mass Spectrometry
LOD	Limit of Detection
LOR	Limit of Reporting
MPA	Methylphosphonic Acid
MS	Mass Spectrometry (or Mass Spectrometer)
m/z	Mass-to-Charge
N-Me DEA	N-methyl Diethanolamine
OPCW	Organisation for the Prohibition of Chemical Weapons
PMPA	Pinacolyl Methylphosphonic Acid
ppm	Parts Per Million
R ²	Correlation Coefficient
RSD	Relative Standard Deviation
SD	Standard Deviation
S/N	Signal-to-Noise
TDG	Thiodiglycol
UPLC	Ultra-Performance Liquid Chromatography

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1. Introduction

The Organisation for the Prohibition of Chemical Weapons (OPCW) is responsible for the implementation of the Chemical Weapons Convention (CWC), with member states collectively working to eliminate the threat of chemical weapons. OPCW designated laboratories, which may be engaged to undertake off-site analysis of OPCW-collected samples, are essential for the organisation's ability to enforce its verification regime and to investigate purported incidents involving chemical weapons.¹ In order for a laboratory to become OPCW designated, it must provide evidence of its ability to competently and unambiguously determine the presence of spiked chemical warfare agents (CWAs) and related compounds in a variety of matrices. There are two main criteria that laboratories must satisfy in order to gain OPCW designation, including the accreditation of the laboratory as having a quality system compliant with international standard ISO17025 and capable of analysing relevant chemicals, and at least annual participation in the OPCW's proficiency testing program with an A-grade majority maintained across every three tests in which the laboratory has participated.

The OPCW proficiency test usually involves six samples, provided in environmental matrices such as soil, water, wipes, or some other solid or semi-solid matrix². Samples may be spiked or blank, with the total number of reportable spiking chemicals ranging from four to nine chemicals for the six samples altogether. After sample receipt, laboratories have 15 calendar days in which to analyse the samples, and provide the results to the OPCW in the form of a report. Consistent results from at least two analytical techniques, often Gas Chromatography-Mass Spectrometry with Electron Impact (GC-MS[EI]), Gas Chromatography-Mass Spectrometry with Chemical Ionisation (GC-MS[CI]) or Liquid Chromatography-Mass Spectrometry (LC-MS), must be provided to support each identification reported.

Defence Science and Technology Group (DST), seeking to achieve OPCW designation, participates regularly in the OPCW proficiency testing program. Given that samples received as part of the program are typically spiked with chemicals of interests at concentrations in the range 1 to 10 ppm,² it is essential that instruments in the Chemical Agents Analysis Facility (CAAF) can detect these chemicals within this concentration range. Accordingly, in order to include developed LC-MS method in future proficiency tests and thereby increase the range of techniques from which data may be reported, the instrument's capacity to detect and report on compounds of interest down to at least 1 ppm needed to be verified. This paper documents the work undertaken in this verification.

The compounds assessed herein include the hydrolysis degradation products of the nerve agents tabun (GA), sarin (GB), soman (GD), GE, cyclosarin (GF) and VX as well as sulphur mustard (HD) and nitrogen mustard (HN2). These are tabulated below:

Table 1. *Hydrolysis products of CWAs*

CWA	Hydrolysis Product
GA	O-Ethyl-N,N-dimethylamido Phosphoric Acid (EDMAP)
GB	Isopropyl Methylphosphonic Acid (IMPA), Methylphosphonic Acid (MPA)
GD	Pinacolyl Methylphosphonic Acid (PMPA), MPA
GE	Ethylphosphonic Acid (EPA)
GF	Cyclohexyl Methylphosphonic Acid (CMPA), MPA
VX	Ethyl Methylphosphonic Acid (EMPA)
HD	Thiodiglycol (TDG)
HN2	N-methyl Diethanolamine (N-Me DEA)

2. Methods

2.1. Sample Preparation

Solutions of the nine hydrolysis products shown in Table 1 were prepared in triplicate for analysis at six concentration levels: 0.25, 0.50, 1, 2.50, 5 and 10 ppm. These solutions were prepared from primary solutions at 1000 ppm, first being diluted 100-fold to 10 ppm. The 10 ppm solution was then used to prepare the 5 ppm and 1 ppm solutions, with each of these then being used in serial dilutions to prepare the remaining concentration levels noted above. Three blanks were also prepared. The series was used for the construction of calibration curves to determine linearity and estimate the limits of detection (LOD) for each compound. It should be noted that the method is intended for qualitative use, and only an indicative LOD was sought.

2.2. LC-MS Method

Analyses were performed using an Agilent Technologies 1290 Infinity Liquid Chromatograph (LC) together with an Agilent Technologies 6460 Triple Quadrupole Mass Spectrometer detector (MS). The method conditions are described below.

2.2.1. LC Conditions

2 µL of each sample was injected onto a Waters Acquity UPLC HSS T3 Column/C-18 (150 mm x 2.1 mm x 1.8 µm), heated to 50 °C, at a flow rate of 500 µL/min. The following eluents were used: (A) 0.05% formic acid in water, and (B) 0.05% formic acid in acetonitrile. The method used a gradient elution program, holding at 100% A for the first two minutes, before ramping down to 10% A over the following 15 minutes, and being held for 5 minutes at a high organic composition. Subsequently, from 22 to 23 minutes, the gradient returned to starting conditions of 100% A.

2.2.2. MS Conditions

The MS was run in positive electrospray ionisation mode, with a capillary voltage of 4.0 kV. Analyses were performed using full-scan mode, monitoring the mass-to-charge (m/z) range 50-650.

3. Results and Discussion

Results were extracted from the data files using Agilent Technologies Mass Hunter Qualitative Analysis software. For each dilution series, the extracted ion chromatograms (EICs) for the relevant compounds were manually integrated, and the area and S/N of each peak calculated by the software. Areas were plotted against concentration in Microsoft Excel to determine linearity over the range assessed (plots are displayed in *Appendix B*).

Table 2. Results of LC-MS analysis of selected CWA hydrolysis products

Compound	Mass-to-Charge Ratio (m/z)	Retention Time (min) (mean \pm SD)	Linearity (R ²)		LOD (ppm)	
			0.25 - 5 ppm	0.25 - 10 ppm	Calculated from 0.25 - 5 ppm Curve	Based on S/N Values
MPA	97	0.846 \pm 0.005	0.9871	0.9923	0.680	~ 0.50
EDMAP	154	1.332 \pm 0.045	0.9911	0.9786	0.598	< 0.25
IMPA	139	4.351 \pm 0.008	0.9984	0.9981	0.225	\leq 0.25
PMPA	361	7.794 \pm 0.036	0.9956	0.9973	0.376	< 0.25
EPA	111	1.117 \pm 0.004	0.9991	0.9959	0.172	< 0.25
CMPA	179	7.110 \pm 0.056	0.9930	0.9976	0.483	0.25 - 0.50
EMPA	125	1.423 \pm 0.180	0.9984	0.9990	0.229	< 0.25
TDG	105	3.183 \pm 0.058	0.9949	0.9861	0.412	< 0.25
N-Me DEA	120	0.789 \pm 0.005	0.9951*		0.201	< 0.25

* Note that N-Me DEA was only linear in the range 0.25 - 2.5 ppm

3.1. Linearity

In the above table, the linear ranges proposed are those which had a correlation coefficient (R²) value of greater than 0.95. Though a quantitative method would typically require R² values of at least 0.99, this method is for qualitative purposes only, and the analysis was carried out without an internal standard to correct for experimental variation. Therefore, an R² value of greater than 0.95 is fit-for-purpose in this instance. All compounds analysed showed acceptable linearity over the range 0.25 to 10 ppm, except for N-Me DEA, which was only linear to an acceptable level up to 2.5 ppm, without correction by an internal standard. This is possibly a result of changing ionic dynamics as the concentration increases.

3.2. Limit of Detection

Despite most compounds showing acceptable linearity for the whole concentration range assessed, the LOD calculations were only performed on the lower half of the curve, from 0.25 to 5 ppm, as the results obtained focusing on this range were more representative of what the LOD would be based on the S/N values observed. Calculation of the LOD from the calibration curves was performed using $LOD = 3 \times$ (standard error of the curve/slope of the curve). Contrastingly, estimation of the LOD from S/N was achieved based on the principle that the LOD is the point where the S/N for a chromatographic peak is equal to three. Average S/N values are displayed in Table 3 below. From the S/N, given an average S/N of 4.0 for MPA at 0.5 ppm, the LOD is approximately equal to 0.5 ppm. Though, a peak was detected in two out of three of the 0.25 ppm samples of MPA, these peaks had a S/N of less than three, and are by definition below the LOD. When calculated from the curve, the LOD for MPA is 0.680 ppm, which is comparable. Similarly, for IMPA, the S/N at 0.25 ppm is only 6.0, indicating that the LOD is close to, but slightly less than, 0.25 ppm, which is in agreement with the calculated LOD of 0.225 ppm based on the curve. CMPA has a S/N of 11.6 at 0.5 ppm, but was not detected at 0.25 ppm, so its LOD would be in the range 0.25 to 0.5 ppm. Again, the LOD calculated from the curve sits in this range at 0.483 ppm. The S/N at 0.25 ppm for EDMAP, PMPA, EPA, EMPA, TDG and N-Me DEA is significantly greater than three, indicating that the LOD for these compounds would be below 0.25 ppm. Although the curve calculated LODs for EPA, EMPA, and N-Me DEA are all in agreement with this, those for EDMAP, PMPA, and TDG are all greater than 0.25 ppm, while remaining under the 1 ppm threshold. The reasons for this could include the inclusion of a higher concentration point at 5 ppm, which may skew the trendline slightly at lower concentrations, whilst the fact that trendlines assume a constant error across the data set could also lead to overestimation of the LOD.

3.3. Limit of Reporting (based on OPCW criteria)

Another important aspect relating to S/N is that for OPCW reporting purposes, chromatographic peaks reported must have a S/N of greater than five. Based on the S/N values in Table 3, this limit of reporting (LOR) is in most cases satisfied for all compounds at the same concentration level examined for which the requirement for LOD is met, except for MPA. In this instance, as the S/N at 0.5 ppm is 4.0, this is approximately at or slightly above the LOD, whilst the LOR, requiring a S/N of greater than five, would be between 0.5 and 1 ppm, with 1 ppm the first concentration level of those assessed to satisfy this criterion. Regardless, MPA remains detectable at reportable levels of at least 1 ppm.

3.4. Repeatability

Variability of the error in instrument response is demonstrated in Table 4, which depicts the average area peak area for each compound at each concentration level, with percent relative standard deviation (%RSD) included. While the %RSD for N-Me DEA across all concentration levels falls in a narrow range of 0.3 to 2.0%, the %RSD for CMPA ranges from 0.8 to 12.2%, showing that there can be significant variation in error across a curve.

Despite this, all concentration levels for the majority of compounds were found to have %RSD of less than 10%, except for CMPA, for which all concentration levels had a %RSD below 15%. These results demonstrate a satisfactory level of repeatability.

OPCW reporting also requires that retention time of the chemical peak be with ± 0.2 min of the corresponding peak of the reference chemical run under similar conditions. Therefore, the repeatability of the retention time was also assessed, with the average retention time and its standard deviation (SD) shown in Table 2 above. All compounds were found to have retention times with a SD of less than ± 0.2 min, and were thus repeatable according to OPCW standards.

Table 3. Average S/N of CWA hydrolysis products at various concentration levels

Compound	Average S/N (# /3 with S/N > 3)					
	0.25 ppm	0.5 ppm	1 ppm	2.5 ppm	5 ppm	10 ppm
MPA	1.6 (0)	4.0 (3) ^c	8.9 (3)	48.2 (2) ^b	130.0 (3)	327.3 (3)
EDMAP	54.9 (2) ^b	95.1 (3)	156.4 (3)	138.1 (2) ^b	157.6 (3)	108.3 (3)
IMPA	6.0 (3)	17.5 (3)	54.5 (3)	153.8 (3)	305.6 (3)	684.2 (3)
PMPA	34.1 (3)	143.7 (3)	676.9 (3)	4434.1 (3)	9151.6 (3)	16738.7 (3)
EPA	15.3 (3)	27.8 (3)	42.9 (3)	85.0 (3)	135.5 (3)	158.3 (3)
CMPA	N/A (0)	11.6 (3)	51.4 (3)	322.1 (3)	883.3 (3)	2242.2 (3)
EMPA	19.3 (3)	35.9 (3)	70.3 (3)	98.4 (3)	119.4 (3)	403.9 (3)
TDG	12.7 (3)	22.2 (2) ^b	39.9 (3)	82.7 (3)	117.9 (3)	172.0 (3)
N-Me DEA	284.9 (3)	284.9 (3)	282.7 (3)	279.8 (3)	330.3 (3)	284.1 (3)

^b Note that for these samples, one point was eliminated as irregular, so the maximum number of replicates was two. Hence 2/2 samples exceeded S/N of 3.

^c Note that though the LOD was satisfied at 0.5 ppm for MPA, the LOR was not satisfied until 1 ppm.

Table 4. Average instrument response for CWA hydrolysis products at various concentration levels

Compound	Average Peak Area \pm %RSD					
	0.25 ppm	0.5 ppm	1 ppm	2.5 ppm	5 ppm	10 ppm
MPA	2.633x10 ⁵ \pm 6.2%	5.596x10 ⁵ \pm 3.0%	1.455x10 ⁶ \pm 9.5%	6.104x10 ⁶ \pm 6.2%	1.052x10 ⁷ \pm 3.4%	2.450x10 ⁷ \pm 5.4%
EDMAP	2.886x10 ⁶ \pm 2.3%	5.846x10 ⁶ \pm 3.3%	1.096x10 ⁷ \pm 3.6%	2.977x10 ⁷ \pm 1.1%	4.928x10 ⁷ \pm 0.9%	7.899x10 ⁷ \pm 3.4%
IMPA	4.615x10 ⁵ \pm 5.2%	1.393x10 ⁶ \pm 4.5%	4.082x10 ⁶ \pm 4.2%	1.195x10 ⁷ \pm 0.5%	2.346x10 ⁷ \pm 2.2%	4.471x10 ⁷ \pm 0.4%
PMPA	4.944x10 ⁵ \pm 1.5%	1.917x10 ⁶ \pm 3.6%	9.885x10 ⁶ \pm 5.2%	4.605x10 ⁷ \pm 2.0%	1.013x10 ⁸ \pm 3.1%	1.956x10 ⁸ \pm 2.0%
EPA	2.051x10 ⁶ \pm 6.0%	4.606x10 ⁶ \pm 2.8%	1.007x10 ⁷ \pm 1.5%	2.813x10 ⁷ \pm 3.0%	5.845x10 ⁷ \pm 2.1%	1.060x10 ⁸ \pm 3.0%
CPA	N/A	2.728x10 ⁵ \pm 12.2%	1.253x10 ⁶ \pm 8.6%	7.533x10 ⁶ \pm 10.8%	1.908x10 ⁷ \pm 0.4%	4.301x10 ⁷ \pm 0.8%
EMPA	1.487x10 ⁶ \pm 4.2%	3.036x10 ⁶ \pm 0.4%	6.412x10 ⁶ \pm 5.2%	1.992x10 ⁷ \pm 1.8%	4.190x10 ⁷ \pm 1.9%	8.190x10 ⁷ \pm 1.8%
TDG	2.651x10 ⁶ \pm 5.6%	5.134x10 ⁶ \pm 3.3%	9.910x10 ⁶ \pm 1.8%	2.220x10 ⁷ \pm 0.5%	3.891x10 ⁷ \pm 0.7%	6.440x10 ⁷ \pm 3.1%
N-Me DEA	3.751x10 ⁷ \pm 2.0%	5.813x10 ⁷ \pm 0.7%	9.443x10 ⁷ \pm 1.2%	1.811x10 ⁸ \pm 1.4%	2.637x10 ⁸ \pm 1.6%	3.553x10 ⁸ \pm 0.3%

4. Conclusion

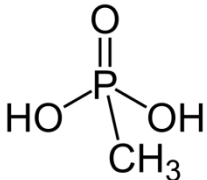
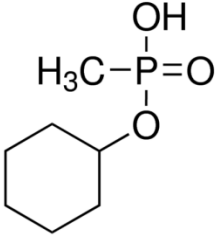
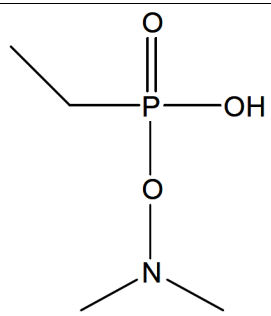
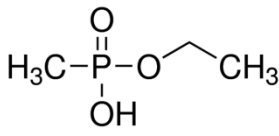
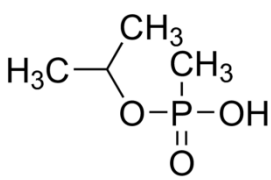
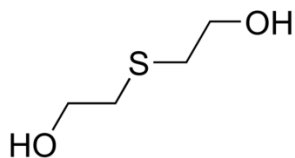
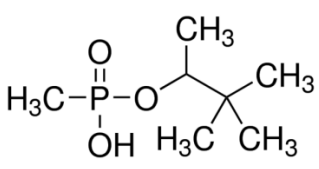
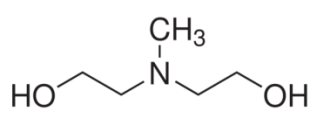
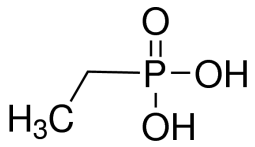
Overall, it was confirmed that the full-scan method developed on the LC-MS is fit-for-purpose for reporting in OPCW proficiency testing, with all compounds assessed as detectable and reportable below the requisite 1 ppm threshold, and with retention times and instrument response satisfactorily repeatable. Furthermore, eight of the nine compounds also showed a high degree of linearity over the 0 to 10 ppm range, with N-Me DEA the exception, which was only linear to 2.5 ppm. As a result, the method was able to be successfully employed in the reporting of spiked chemicals in the 43rd Official OPCW Proficiency Test.

5. References

1. OPCW Designated Laboratories. <https://www.opcw.org/our-work/non-proliferation/designated-laboratories/> (accessed August 2018).
2. Konopski, L.; Liu, P.; Wuryani, W.; Śliwakowski, M., OPCW Proficiency Test: A Practical Approach Also for Interlaboratory Test on Detection and Identification of Pesticides in Environmental Matrices. *The Scientific World Journal* **2014**, 2014, 542357.

Appendix A Chemical Structures

Table 5 - Chemical Structures of CWA Hydrolysis Products

Structure	Name/ Description	Structure	Name/ Description
	Methylphosphonic Acid (MPA) <i>Degradation product of various Nerve Agents</i>		Cyclohexyl Methylphosphonic Acid (CMPA) <i>Degradation product of various GF</i>
	O-Ethyl-N,N-dimethylamido Phosphoric Acid (EDMAP) <i>Degradation product of GA</i>		Ethyl Methylphosphonic Acid (EMPA) <i>Degradation product of VX</i>
	Isopropyl Methylphosphonic Acid (IMPA) <i>Degradation product of GB</i>		Thiodiglycol (TDG) <i>Degradation product of HD</i>
	Pinacolyl Methylphosphonic Acid (PMPA) <i>Degradation product of GD</i>		N-methyl Diethanolamine (N-Me DEA) <i>Degradation product HN2</i>
	Ethylphosphonic Acid (EPA) <i>Degradation product of GE</i>		

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Appendix B Calibration Curves

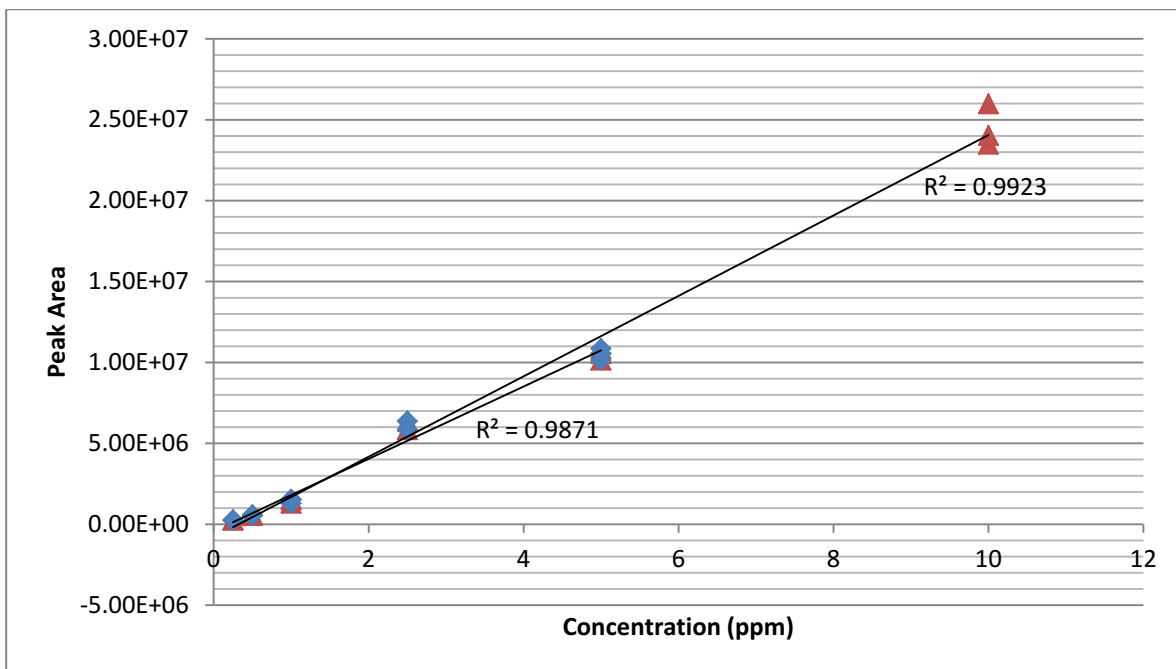


Figure 1. Plots of Concentration versus Peak Area for MPA across the ranges 0.25 – 5 ppm and 0.25 – 10 ppm

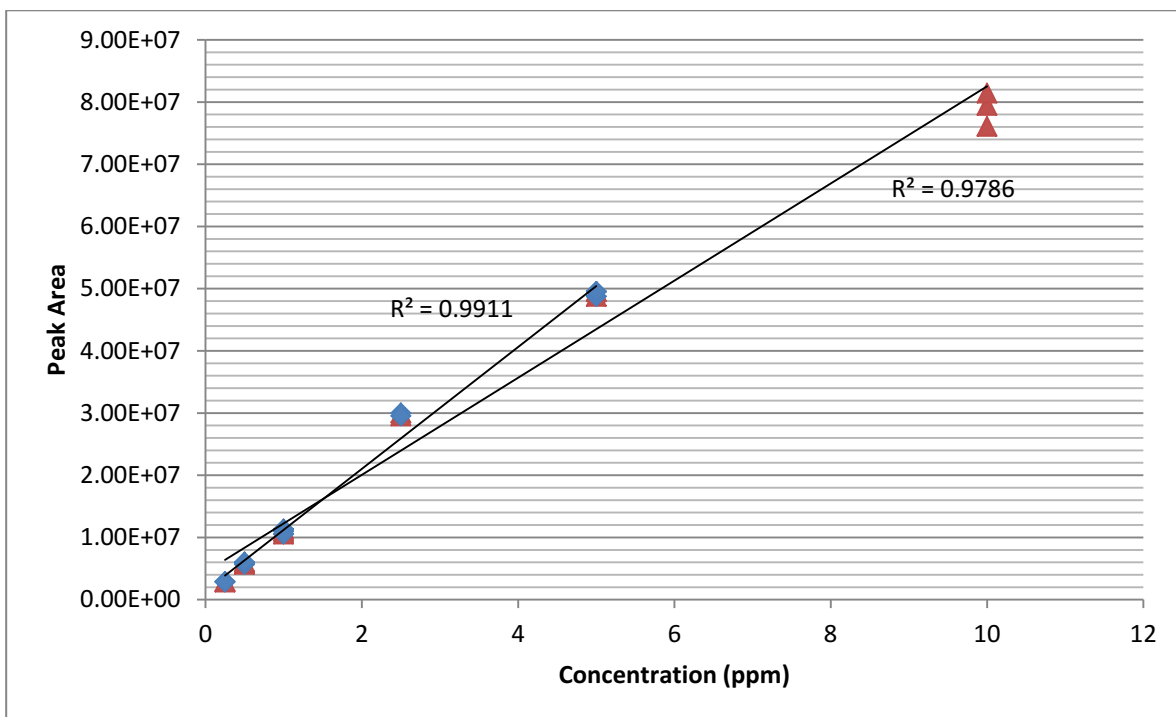


Figure 2. Plots of Concentration versus Peak Area for EDMAP across the ranges 0.25 – 5 ppm and 0.25 – 10 ppm

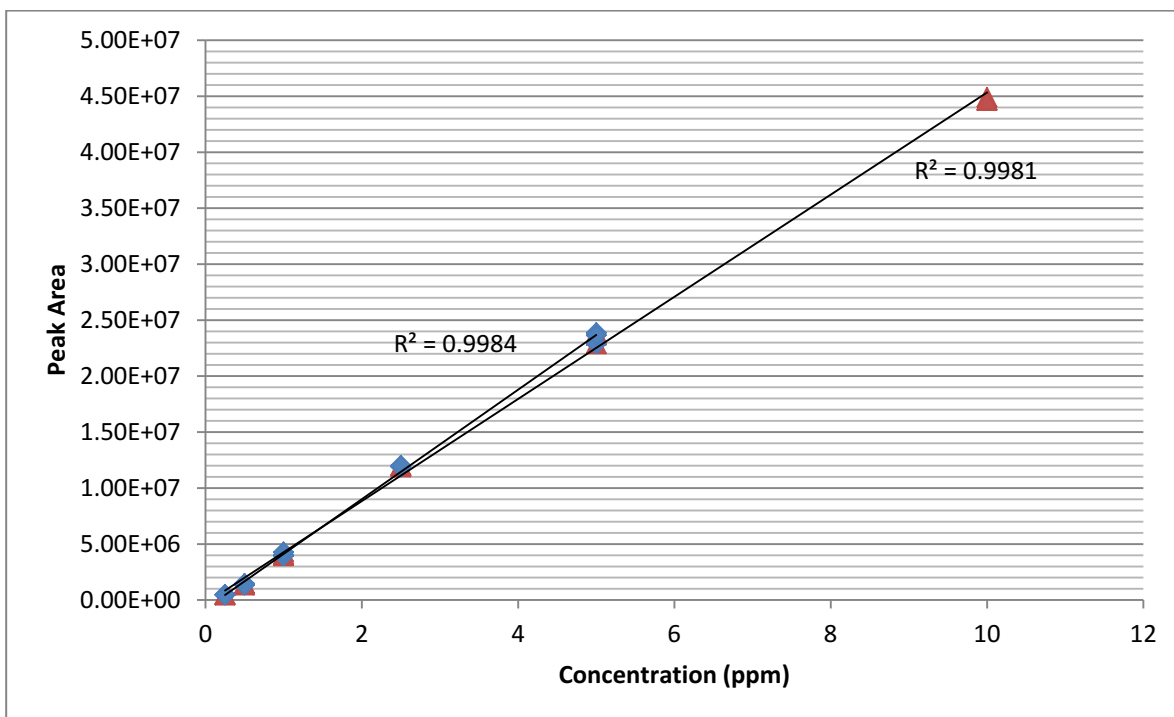


Figure 3. Plots of Concentration versus Peak Area for IMPA across the ranges 0.25 - 5 ppm and 0.25 - 10 ppm

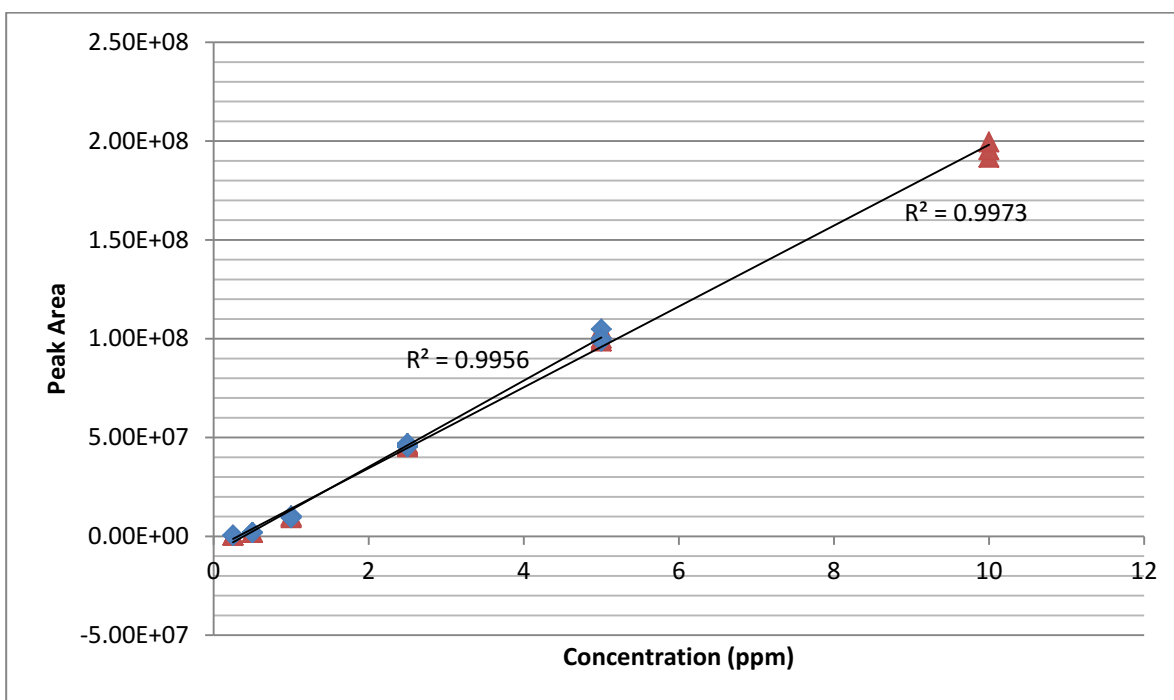


Figure 4. Plots of Concentration versus Peak Area for PMPA across the ranges 0.25 - 5 ppm and 0.25 - 10 ppm

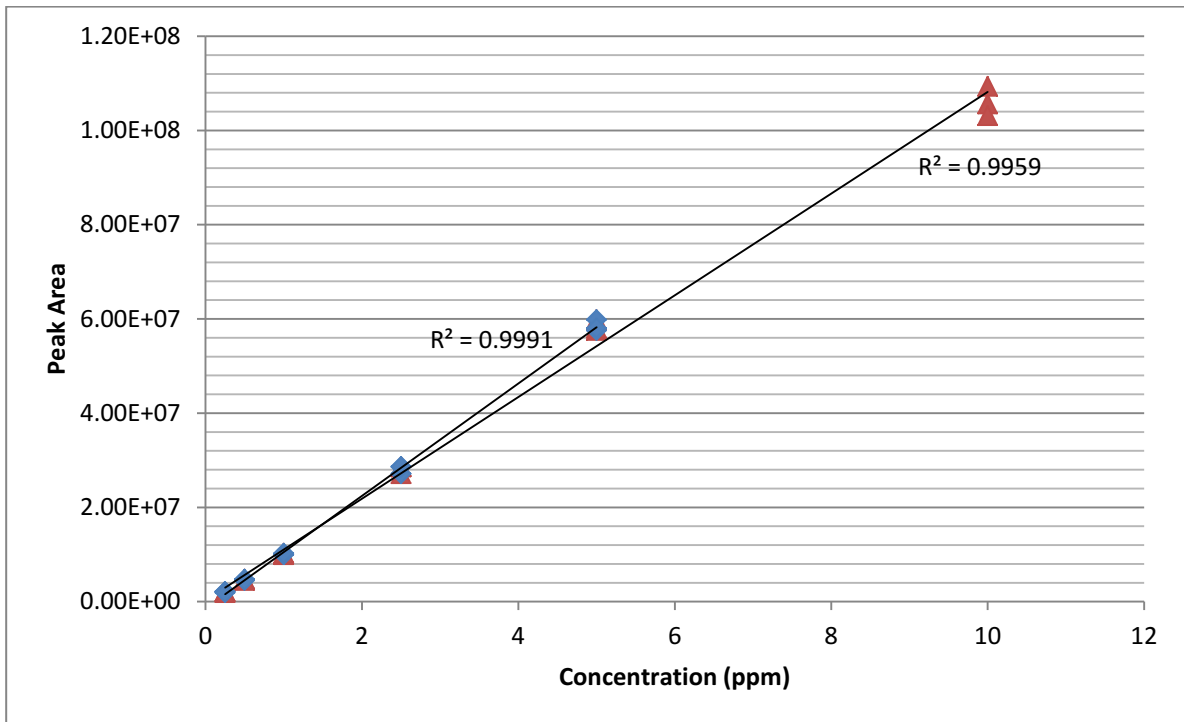


Figure 5. Plots of Concentration versus Peak Area for EPA across the ranges 0.25 – 5 ppm and 0.25 – 10 ppm

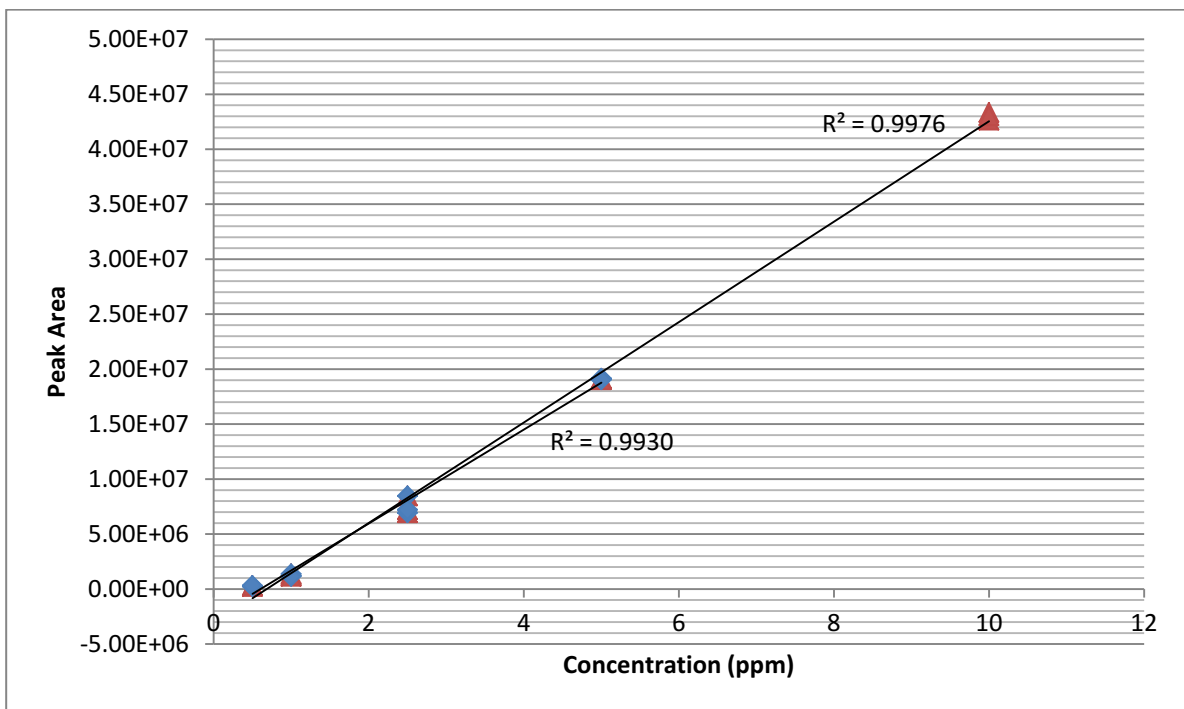


Figure 6. Plots of Concentration versus Peak Area for CMPA across the ranges 0.25 – 5 ppm and 0.25 – 10 ppm

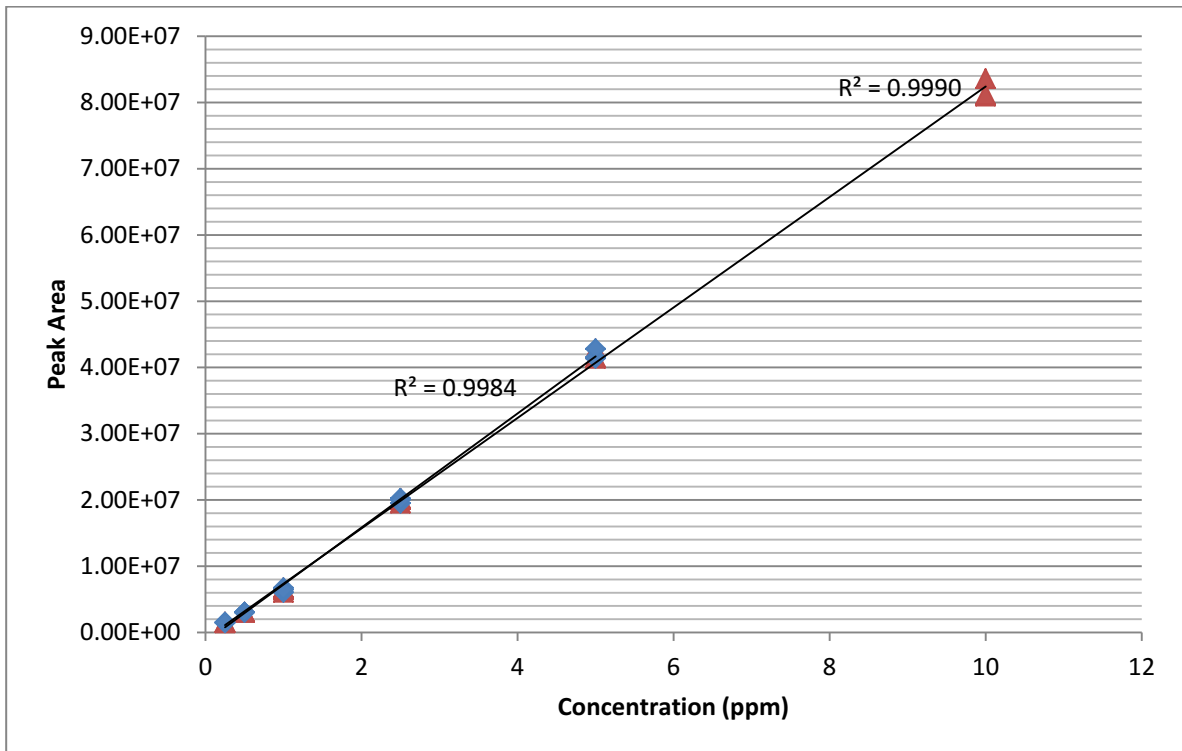


Figure 7. Plots of Concentration versus Peak Area for EMPA across the ranges 0.25 – 5 ppm and 0.25 – 10 ppm

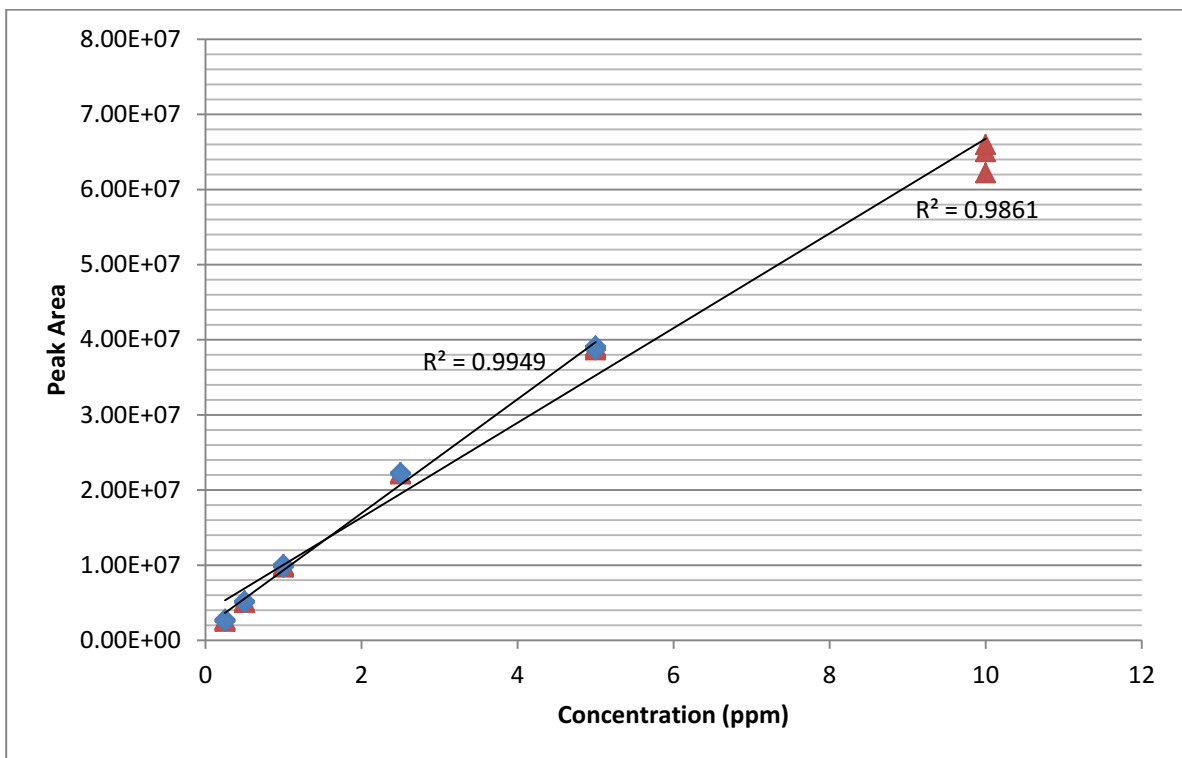


Figure 8. Plots of Concentration versus Peak Area for TDG across the ranges 0.25 – 5 ppm and 0.25 – 10 ppm

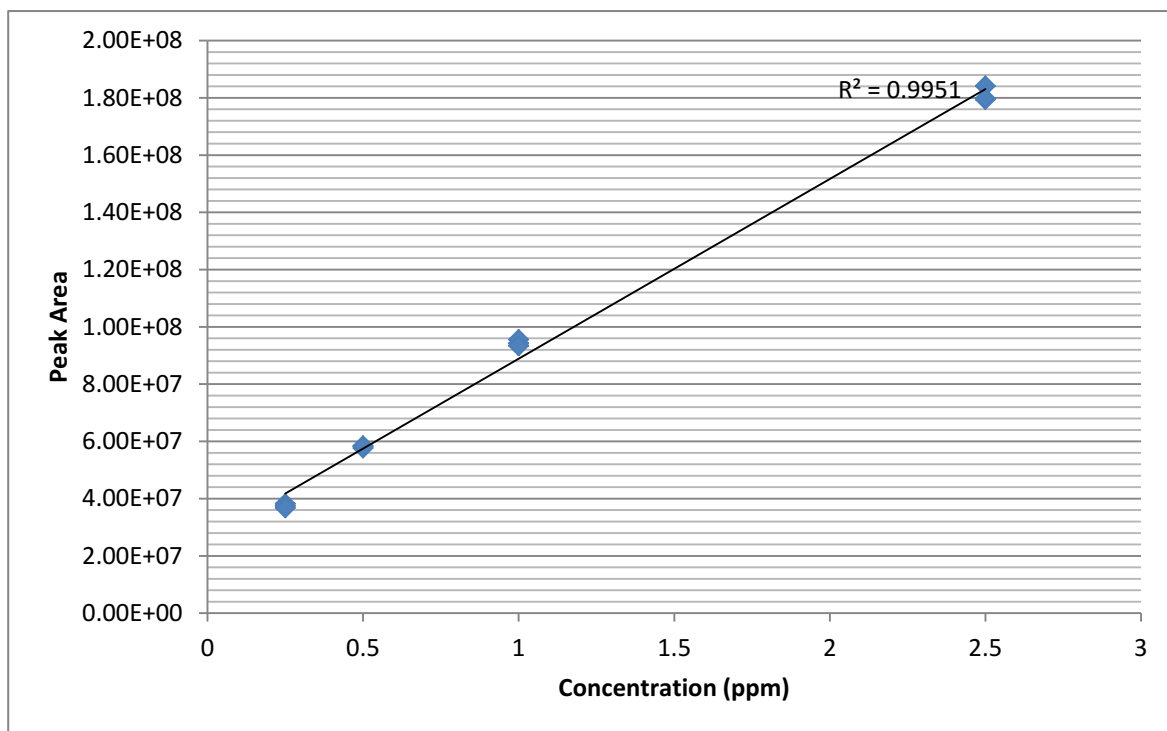


Figure 9. Plot of Concentration versus Peak Area for N-Me DEA in the range 0 – 2.5 ppm

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17. ABSTRACT Defence Science and Technology (DST) Group is working towards becoming a designated laboratory for the Organisation for the Prohibition of Chemical Weapons (OPCW), and thus participates in its proficiency testing program. DST has typically relied on a combination of data from Gas Chromatography-Mass Spectrometry with Electron Impact (GC-MS[EI]) and Gas Chromatography-Mass Spectrometry with Chemical Ionisation (GC-MS[CI]) for reporting purposes, however it has recently implemented a Liquid Chromatography-Mass Spectrometry (LC-MS) method as a third technique to provide additional information where required. Given that samples received as part of the program may be spiked with compounds at concentrations as low as 1 ppm, this document reports the work undertaken in ascertaining that the limit of detection (LOD) of the LC-MS method is below this threshold, and that the method is overall fit for application in OPCW proficiency tests. The results of the work demonstrate that for all compounds assessed, the LOD is below 1 ppm and the method is acceptably repeatable so that it may be used for reporting in OPCW proficiency tests.		

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