



**NEICVP1478E02
Replacement Report**

**NEIC CIVIL INVESTIGATION REPORT
GMAP R7 FY2022
St. Louis, Missouri**

Investigation Dates:
September 12-15, 2022

Project Manager

Analytical Project Manager

Bradley Venner

Richard Helmich

Authorized for Release by:

Martha Hamre, Acting Field Branch Manager, NEIC

Report Prepared for:
Tracey Casburn, EPA Region 7
11201 Renner Boulevard
Lenexa, Kansas 66219

NATIONAL ENFORCEMENT INVESTIGATIONS CENTER
P.O. Box 25227
Building 25, Denver Federal Center
Denver, Colorado 80225

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and provides a clear indication of the end of this report.**

INVESTIGATION OVERVIEW

This report (NEICVP1478E02) replaces the following U.S. Environmental Protection Agency (EPA) National Enforcement Investigations Center (NEIC) report in its entirety: NEICVP1478E01 (December 2022). This replacement was necessary to correct the following: isobutane was used to calibrate the photoionization detector (PID); the previous report stated that isobutylene was used. Total volatile organic compound (VOC) concentrations determined by the PID in this report have been recalculated and are reported as isobutylene equivalent for consistency with previous NEIC GMAP reports.

At the request of U.S. Environmental Protection Agency (EPA) Region 7 (Region), the EPA National Enforcement Investigation Center (NEIC) performed an EPA draft Other Test Method (OTM) 33, *Geospatial Measurement of Air Pollution, Remote Emissions Quantification (GMAP)*, survey outside the fence line of various stationary sources of air pollution in or nearby St. Louis, Missouri. GMAP surveys may help regulated entities, the government, and the public locate elevated pollutant concentrations on or near selected facilities (concentration mapping) and identify potential emission sources that may contribute to these elevated pollutant concentrations (emission source characterization). The Region provided guidance to NEIC on selecting facilities of interest within the survey area.

The GMAP survey was conducted from September 12-15, 2022. Measured air pollutants included methane (CH₄) and hydrogen sulfide (H₂S) using a cavity ring-down spectrometer (CRDS); total VOCs using a PID; and benzene (BEN), toluene (TOL), m-xylene (XYM), and p-xylene (XYP) (collectively referred to as BTX) using a differential ultraviolet (UV) absorption spectrometer (DUVAS). A forward-looking infrared (FLIR) camera capable of visualizing gaseous volatile organic compounds was used to locate potential emission sources when elevated readings were detected. Air samples were collected at selected locations into 1.4-liter (L) evacuated canisters, following the NEIC operating procedure *Air Sampling*, NEICPROC/11-008. Canisters were analyzed for volatile organic compounds following EPA Compendium Method TO-15A using a gas chromatograph with a time-of-flight mass spectrometer (GC-TOFMS) and for light hydrocarbon gases following ASTM Method D7833 as guidance using a gas chromatograph with a flame ionization detector (GC-FID).

Field measurement results from the CRDS, PID, and DUVAS instruments were processed into files in the Keyhole Markup Language (KML) format and are provided in **Appendix A**; FLIR camera video files in MP4 format that were collected by NEIC field team members are provided in **Appendix B**.

METHODOLOGY

General GMAP methodology is described in EPA draft test method OTM33 (version 1.2). Sub-method OTM 33A (version 1.3) provides detailed method requirements, performance metrics, and method quality indicators. This section of the report provides details about NEIC's implementation of this method.

PERSONNEL

Table 1 lists the project team members.

Table 1. PROJECT TEAM MEMBERS		
Name	Organization	Role
Bradley Venner	EPA-NEIC	Project manager
Richard Helmich	EPA-NEIC	Principal GMAP operator
Philip Myers	EPA-NEIC	Field team member
Cynthia Shafer	EPA-NEIC	Field team member
Amy Harvey	EPA-NEIC	Laboratory team member
REGIONAL AND OTHER CONTACTS:		
Joe Terriquez	EPA Region 7, Enforcement and Compliance Assurance Division, Air Branch	Field team member

INSTRUMENTATION

Table 2 lists the instruments used in the survey.

Table 2. GMAP ANALYTES AND INSTRUMENTATION				
Analyte/Measurand	Instrument	Manufacturer	Model	Serial No.
CH ₄ and H ₂ S	CRDS	Picarro	G2204	3227-BFADS2027
Benzene, toluene, m-xylene, p-xylene, sulfur dioxide	DUVAS	DUVAS	DV3000	DV1003
VOCs	PID	Ion Science	MiniPID2-HS	MP3SHLHSCU2
Motion-corrected wind speed and direction	Weather station	AirMar	200WX	3445952
Spatial location	Geographical positioning system	Hemisphere	Crescent R100	0734-4068-003
Imaging, VOCs	Infrared imaging camera	FLIR	GFx320	C12140

CALIBRATION

Calibration verifications for the CRDS, DUVAS, and PID instruments were performed at the beginning and the ending of each working day. Corresponding calibration gases were metered to each instrument from the cylinders through a valved manifold. The calibration gases used in this project were a BTEX mixture for the DUVAS, isobutane for the PID, and H₂S and CH₄ for the CRDS. Detailed descriptions and certificates of analysis (CoAs) of the calibration gases are in the project file. Calibration verifications also included analysis of "ultra-zero air," which contains, at most, only very small quantities of any analyte.

Time periods during the calibration process when relatively constant responses to ultra-zero air and calibration gas inputs were obtained were visually identified by Bradley Venner. **Table 3** summarizes the daily quantitative calibration results. **Appendix C** includes plots of the ultra-zero air and calibration gas responses.

Table 3. SUMMARY OF DAILY CALIBRATION VERIFICATION RESULTS						
Calibration Level (Span or Zero)	Analyte	Units	Calibration Standard Concentration	Average Measured Concentration of Calibration Events	Standard Deviation Between Calibration Events	Pooled Standard Deviation Within Calibration Events
Span	Benzene	ppb	337	275	14.5	3.2
Span	CH ₄	ppm	20	21	0	0.0
Span	H ₂ S	ppb	49420	47737	35.9	96.3
Span	Toluene	ppb	335	287	11.2	5.5
Span	VOCs	ppb	1990	1759	123.0	24.4
Span	m-xylene	ppb	332	274	56.8	17.6
Span	p-xylene	ppb	338	292	3.0	6.1
Zero	Benzene	ppb	0	<0	7.7	3.7
Zero	CH ₄	ppm	0	0	0	0.0
Zero	H ₂ S	ppb	0	4	1.5	2.2
Zero	Toluene	ppb	0	<0	5.5	4.3
Zero	VOCs	ppb	0	<0	65.7	22.7
Zero	m-xylene	ppb	0	<0	30.4	17.5
Zero	p-xylene	ppb	0	2	4.2	2.2

As noted above, the PID for this project was calibrated with isobutane, and VOC concentration raw data were output as ppb isobutane equivalent. The more common calibration gas used for a PID is isobutylene (isobutene), which has been used to calibrate the GMAP’s PID in previous surveys. For consistency with previous reporting, all VOC concentrations provided in this replacement report and appendices have been converted from ppb isobutane equivalent to ppb isobutylene equivalent, unless otherwise noted. The conversion from VOC concentration in ppb isobutane equivalent to VOC concentration in ppb isobutylene equivalent requires division by the isobutane response factor, which is equal to 8 for the 10.6 eV lamp that was used in the vehicle.¹

The response from the ultra-zero air analysis was used to calculate a method detection limit (MDL) for each analyte. The MDL was calculated using the largest standard deviation for the zero standard from all valid calibration files multiplied by 2.39, the 99% quantile of a t-distribution with 60 degrees of freedom. A minimum detectable concentration (also referred to as an upper control limit or UCL) was calculated by multiplying the MDL by 2.2. Daily UCL and MDL values were calculated by adding the daily average concentration value for the zero

¹ Ion Science. “Technical/Application Article 02, ION Science PID Gas Sensor Response Factors” V. 1.0, Oct. 13, 2020.

standard, if positive. **Table 4** shows the calculated MDL and UCL values for the DUVAS analytes and for hydrogen sulfide. Methane and VOC are not shown since fixed mapping scales were used for these analytes.

	Benzene		Hydrogen sulfide		Toluene		m-Xylene		p-Xylene	
Largest Standard Deviation	6		3		5		26		3	
MDL¹	13		7		13		62		6	
UCL²	29		16		28		137		14	
Day	<i>Min</i>	<i>Max</i>	<i>Min</i>	<i>Max</i>	<i>Min</i>	<i>Max</i>	<i>Min</i>	<i>Max</i>	<i>Min</i>	<i>Max</i>
September 12, 2022	22	67	11	36	14	57	62	275	12	39
September 13, 2022	13	58	11	35	13	56	62	275	10	36
September 14, 2022	13	58	11	35	13	56	62	275	10	39
September 15, 2022	17	62	15	39	13	56	90	302	16	43
¹ MDL: method detection limit										
² UCL: upper control limit										
NOTE: All concentrations are in parts per billion (ppb)										

DATA MANAGEMENT

Instrument operation and data collection were managed by an on-board computer running custom application software, Mobile Emissions Monitoring Software (MEMS), version 6.2.1. Data were recorded from each instrument approximately every second. Data were stored in individual, tab-delimited text files that were started and stopped by the instrument operator. Individual text files are referred to as “mapping runs.” The MEMS software displays selected analytes in real-time, which allows for dynamic vehicle routing based on observed measurements. Canister sampling is also controlled, and the start/stop times are recorded, by MEMS.

Isobutylene-equivalent VOC values were calculated by importing the text files created by MEMS into the R statistical software package, dividing the isobutane-equivalent VOC values by 8, and then exporting the results to a new text file. The R scripts and the exported text files are available in the project file.

Individual text files were processed by the custom application software Google Earth Map Plotter, version 1.7. This software produces KML files that can be opened using geographic information systems such as Google Earth Pro (GEP). **Table 4** provides the calculated mapping scales for each DUVAS analyte and hydrogen sulfide for each day. The minimum mapping scale (green) was set to the daily MDL. The maximum mapping scale (red) was calculated as two times the UCL plus the daily average concentration for the zero standard, if positive. Values greater than maximum mapping scale appear on the maps as proportionally taller red bars. Mapping scales for the VOCs were fixed at a minimum (green) of 200 ppb and a maximum (red)

of 400 ppb. Mapping scales for methane were fixed at a minimum (green) of 2 parts per million (ppm) and a maximum (red) of 4 ppm.

Analyte-specific KML files were produced where the maximum value within the run exceeded the daily UCL. The electronic version of **Appendix A** provides the resulting KML files. **Appendix D** provides directions for using GEP to visualize these KML files.

AIR CANISTER SAMPLING AND LABORATORY ANALYSIS

Prior to deployment for this project, the laboratory team cleaned each canister using at least three nitrogen gas pressurization/evacuation cycles, followed by a clean check analysis for volatile organic contaminants using a GC-TOFMS and a GC-FID, confirming canister cleanliness for both TO-15A and light hydrocarbon analytes. After clean checks were performed, the laboratory team evacuated the canisters to ≤ 3 millitorr (mtorr) and then provided them to the field team for sampling.

The field team collected air canister samples following NEIC procedure *Air Sampling*, NEICPROC/11-008. Canisters were opened for approximately 1 minute. Canisters were opened and closed using the MEMS onboard software, which records the sampling time on the file for the associated mapping ID. **Appendix E** provides information on canister sampling locations, times, associated mapping ID, and sampler. After the sample canisters were collected in the field, they were shipped back to the NEIC laboratory on September 16, 2022, by FedEx, tracking No. 572243903299. Amy Harvey of the laboratory team received the samples on September 19, 2022. **Appendix E** includes a digital copy of the chain of custody form.

Light hydrocarbons analysis was performed first, without diluting the samples. Following the light hydrocarbon analysis, all sample canisters were diluted/pressurized using the static dilution system before TO-15A analysis was performed.

Table 5 summarizes the laboratory team’s analysis of the samples.

Table 5. ANALYTICAL OBJECTIVE, TECHNIQUE, METHOD; ANALYST; AND DATE PERFORMED		
Analytical Objective, Technique, and Method ¹	NEIC Analyst	Dates Performed
Volatile organic compounds: EPA Compendium Method TO-15A by gas chromatography-mass spectrometry (GC-MS) – canister samples	Amy Harvey	Clean checks: August 25-30, 2022 Sample analysis: September 29-October 15, 2022
ASTM Method D7833, Standard Test Method for Determination of Hydrocarbons and Non-Hydrocarbon Gases in Gaseous Mixtures by Gas Chromatography (as guidance)		Clean checks: August 23-29, 2022 Sample analysis: September 20-26, 2022
¹ Data quality summaries, including uncertainty measurements, for all laboratory measurements are maintained in the project file.		

QUALITY ASSURANCE

All environmental measurement activities were performed in accordance with the NEIC quality system. All field sampling, field measurements, and laboratory analyses described in this report were within the scope of NEIC’s ISO/IEC 17025 accreditation issued by the ANSI National Accreditation Board (certificate No. FT-0303), except for the motion-corrected anemometer. The GMAP motion-corrected anemometer cannot be calibrated as a complete system; therefore, the uncertainty of this instrument cannot be verified.

According to the manufacturer, DUVAS-measured compounds are linear to a value of 1,000ppb. The manufacturer has identified the operating range for the PID sensor as 0–3,000ppb isobutylene equivalent. Readings greater than these operating ranges will have a higher uncertainty. The operating ranges for methane and hydrogen sulfide on the CRDS instrument are 0–20ppm and 0–300ppm, respectively.

The AirMar weather station failed to provide wind speed and direction data on approximately 1% of the field measurements.

For several mapping runs, the maximum concentration in the KML file was manually corrected. In these cases, the relevant concentrations are marked with an asterisk in **Appendix F**.

RESULTS

GMAP activities were conducted on 4 days during the investigation period. **Appendix F** provides detailed information of GMAP activities, indexed by mapping run. **Table 6** summarizes the information provided in **Appendix F**, indexed by day. **Appendix E** provides the canister sampling times, locations, and operators in detail. **Appendix B** provides details on FLIR video files. The field logbook contains further information regarding the measurement and sampling activities performed for this project.

Table 6. SUMMARY OF GMAP ACTIVITIES BY DAY			
Day	Map ID	FLIR Video File Name (MOV_####)	Canisters
September 12, 2022	220912_MA01–220912_MA79	0221–0226	3110, 4608, 9498
September 13, 2022	220913_MA01–220913_MA59	0227–0230	277, 533, 2629, 4610, 4615
September 14, 2022	220914_MA01–220914_MA53	0231–0235	515, 531, 2070, 4611, 10024
September 15, 2022	220915_MA01–220915_MA62	0236–0245	852, 3069, 3072

CONCENTRATION MAPPING

Figure 1 shows an overview of the mapping runs. This map indicates all the locations where GMAP data was collected (green lines). Canister sampling locations are marked with a teardrop-shaped red icon. The overnight monitoring location is marked with a yellow cross-shaped icon.

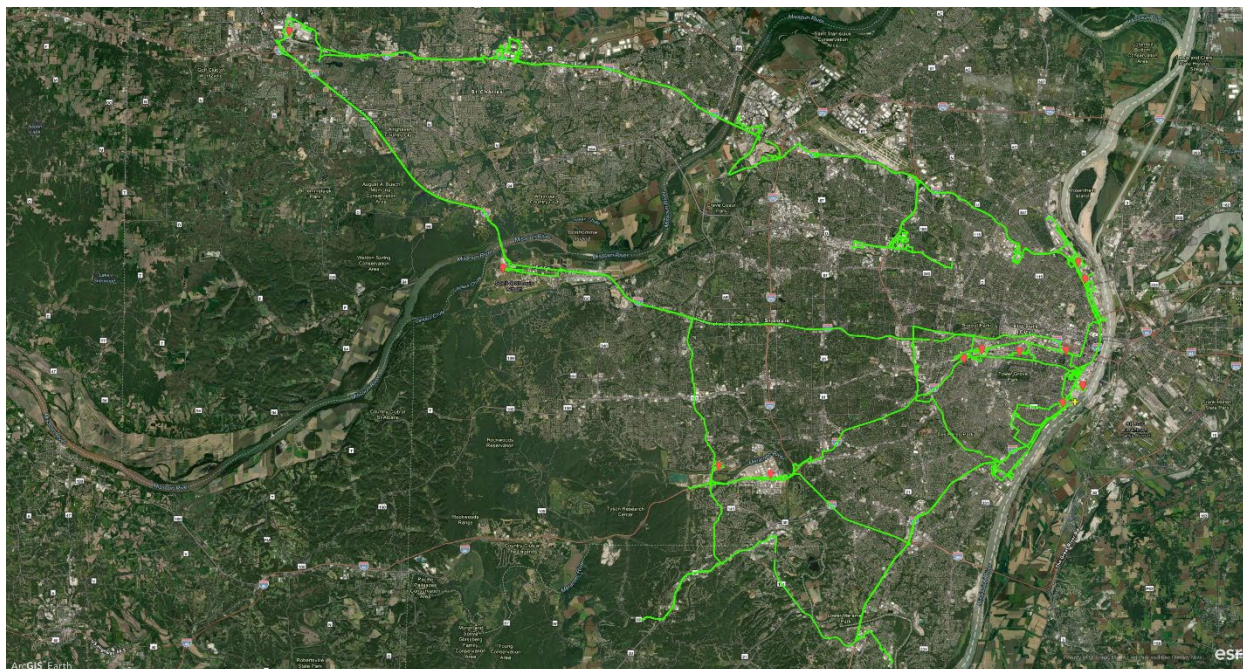


Figure 1. Overview of GMAP mapping runs; St. Louis, Missouri; September 12-15, 2022

NEIC deployed the GMAP vehicle overnight on September 12, 13, and 14, 2022, and collected data during this time period. Pollution bivariate polar plots were developed from this data set for the analytes methane, hydrogen sulfide, and total VOCs using the R package “openair” and the routine “polarPlot.” **Appendix G** shows these plots. These plots illustrate the smoothed average concentration for the pollutant distributed over wind speed and wind direction. Higher wind speeds are further from the center than lower wind speeds. VOC concentrations were normalized by subtracting the overnight minimum value from the VOC concentrations, so that the minimum value was zero. This was done to facilitate comparison of VOC concentrations between days. Wind directions on the map correspond to the direction that the wind is blowing from. The smoothed average concentration is shown on the color scale shown on each figure. Monitoring began at approximately 7:00 p.m. each evening and concluded at 8:00 a.m. the following morning. **Table 7** shows the mapping IDs used for each overnight monitoring event and each event’s start and end times.

For the overnight monitoring from September 14-September 15, 2022, the VOC plot is consistent with sources predominantly from the north-east for wind speeds higher than 2 meters/second (m/s), with some elevated readings from the north. For the overnight monitoring from September 12-September 13, 2022, the hydrogen sulfide plot is consistent with a north-northwest source for wind speeds higher than 2 m/s.

Table 7. MAPPING IDS USED IN OVERNIGHT MONITORING			
Overnight	Map_ID	Start Time (24 hour)	End Time (24 hour)
September 12-13, 2022	220912_MA79	19:10	0:09
	220913_MA01	0:10	5:00
	220913_MA02	5:00	7:51
	220913_MA03	7:52	8:00
September 13-14, 2022	220913_MA59	19:00	0:01
	220914_MA01	00:01	8:00
September 14-15, 2022	220914_MA53	19:00	8:00

SOURCE CHARACTERIZATION

NEIC field team members used FLIR infrared imaging cameras to attempt to locate upwind sources of elevated concentrations detected during concentration mapping activities. These videos are summarized in the table in **Appendix B**, with the date and time of collection, the equipment used, the thermographer, the associated facility, and the mapping ID. The electronic version of this report includes the digital versions of the collected videos.

NEIC collected a total of 16 canister samples of air from 14 distinct locations during the GMAP survey during potentially elevated concentration events or as field blanks. **Appendix E** lists the canister identification number, sampling time, and GPS locations where each canister sample was collected.

Table 8 summarizes source characterization activities undertaken during the survey. This table shows the mapping ID associated with canister sampling or FLIR video collection, along with the maximum recorded field measurement concentration during the mapping run.

Appendix H and **Appendix I** summaries air canister analysis results for TO-15A analytes and light hydrocarbon analytes. **Appendix J** shows detailed analytical results for the TO-15A analytes, including compounds that were not detected and their quantitation limits. **Appendix K** contains a summary of analytes that were tentatively identified (TICs) in the TO-15A analysis. The identification is based solely on a spectrum and/or interpretation of the spectrum to a library database (e.g., Wiley, National Institute of Standards and Technology, in-house), with no analysis of a certified reference standard. The library similarity factor to the library spectrum was > 850, and the peak signal-to-noise was > 4000 for each TIC. The 10 highest response TICs meeting these criteria are reported, unless there were none of any significant response, and they are listed in the elution order as detected by the mass spectrometer.

Table 9 shows a comparison of the average concentration measured with the field instruments during the canister sampling time with the measured concentrations in the air canisters. The average toluene concentration measured by the DUVAS and the average VOC concentration measured by the PID were calculated over the time period where the air canister was open.

Synthetic PID-equivalent VOC readings for the air canisters were calculated by dividing the concentration of each VOC quantitated in the canister by the GC-TOFMS by the response factor for a 10.6 electron volt (eV) UV lamp developed by Ion Science, which is the sensor used in the GMAP vehicle². These average calculations assume a constant flow rate into the canister during the sampling period. The toluene concentration in the canister were measured by the GC-TOFMS.

The toluene values are generally within a factor of 2 between the two methods. The VOC measured by the PID were generally within a factor of 3, with occasionally much larger differences. Some of the discrepancies may be due to the presence of tentatively identified compounds in the canister. **Appendix K** shows the qualitative identity of these compounds and their area counts, but these were not quantified and cannot be included in the synthetic response.

Table 8. SUMMARY OF SOURCE CHARACTERIZATION ACTIVITIES								
Map ID	H ₂ S	BEN	TOL	XYM	XYP	VOC	Can ID/FLIR File Name	Potential Facility
	(ppb)							
220912_MA36	11	13	<DL	<DL	16	95	MOV_0221	PQ Corp
220912_MA44	13	<DL	14	<DL	14	108	MOV_0222	Trans Chemical
220912_MA52	12	13	150	<DL	15	1,526	CAN_4608/MOV_0224, MOV_0225, MOV-2226	Trans Chemical
220912_MA68	12	<DL	13	<DL	13	265	CAN_3110	U.S. Paint
220912_MA78	<DL	<DL	<DL	<DL	11	1,890	CAN_9498	Field Blank
220913_MA06	<DL	<DL	<DL	<DL	8	2,164	CAN_533, CAN_277	Schaeffer Manufacturing
220913_MA07	12	<DL	<DL	<DL	7	5,439	CAN_4610	Schaeffer Manufacturing
220913_MA15	<DL	<DL	<DL	<DL	8	665	MOV_0227	Buckeye Terminal
220913_MA35	11	<DL	<DL	<DL	10	5,928	CAN_4615	Chemisphere
220913_MA36	<DL	<DL	<DL	<DL	11	235	MOV_0229, MOV_0230	Elementis Specialities
220913_MA37	<DL	<DL	63	<DL	11	2,392	CAN_2629	Midwest Industrial Chemical

² Ion Science. "Technical/Application Article 02, ION Science PID Gas Sensor Response Factors" V. 1.0, Oct. 13, 2020.

Table 8. SUMMARY OF SOURCE CHARACTERIZATION ACTIVITIES

Map ID	H ₂ S	BEN	TOL	XYM	XYP	VOC	Can ID/FLIR File Name	Potential Facility
	(ppb)							
220914_MA07	14	<DL	<DL	<DL	10	608	MOV_0231	Kinder Morgan Asphalt Terminal
220914_MA13	<DL	<DL	<DL	<DL	8	4,582	CAN_3070	Millipore Sigma
220914_MA17	<DL	<DL	<DL	<DL	8	147	MOV_0232	Schaeffer Manufacturing
220914_MA21	<DL	<DL	<DL	<DL	8	327	MOV_0233	U.S. Paint
220914_MA23	11	<DL	39	<DL	8	4,217	CAN_10024	Willert Home Products
220914_MA27	<DL	<DL	37	<DL	12	3,799	CAN_531	Specialty Chemical
220914_MA34	<DL	<DL	835	79	113	12,450	CAN_4611	FOL Tape
220914_MA36	12	<DL	<DL	<DL	12	779	MOV_0235	Reichhold Chemical
220914_MA49	<DL	<DL	<DL	<DL	13	2,798	CAN_515	Quality Screw Machine Products
220915_MA31	<DL	<DL	<DL	104	<DL	368	MOV_0236, MOV_0237	Fred Weber Asphalt Plant
220915_MA35	<DL	<DL	19	136	9	3,337	CAN_852, CAN_3072	Euclid Chemical
220915_MA46	<DL	<DL	39	453	98	6,923	CAN_3069	Reichhold Chemicals
220915_MA47	<DL	<DL	21	392	81	3,713	MOV_0238, MOV_0239	Reichhold Chemical
220915_MA57	<DL	<DL	<DL	166	<DL	2,038	MOV_0240, MOV_0241, MOV_0242	JD Streett
220915_MA59	<DL	<DL	<DL	146	16	1,857	MOV_0243	Buckeye Terminal
220915_MA61	<DL	<DL	<DL	110	<DL	1,166	MOV_0244, MOV_0245	Kinder Morgan Asphalt Terminal

Table 9. COMPARISON OF GMAP AND CANISTER CONCENTRATIONS					
Map ID	Canister ID	VOC (ppb)		Toluene (ppb)	
		GMAP	Canister	GMAP	Canister
220912_MA52	4608	863	495	47.9	67
220912_MA68	3110	216	335	<DL	23.4
220912_MA78	9498	76	3.4	<DL	< QL
220913_MA06	277	655	224	<DL	< QL
220913_MA06	533	655	186	<DL	< QL
220913_MA07	4610	2,250	946	<DL	< QL
220913_MA35	4615	549	1,050	<DL	10.7
220913_MA37	2629	2,038	853	47.5	112
220914_MA13	3070	2,975	1,370	<DL	< QL
220914_MA23	10024	2,713	270	21.5	1.2
220914_MA27	531	2,088	919	18.3	67.5
220914_MA34	4611	12,450	24,500	638	1,410
220914_MA49	515	2,025	27	<DL	1.63
220915_MA35	852	654	318	<DL	< QL
220915_MA35	3072	654	280	<DL	< QL
220915_MA46	3069	1,388	461	<DL	< QL

DISCUSSION

GMAP data are best used to screen for areas where further investigation using more traditional inspection and leak detection instruments can help to determine if emissions meet regulatory requirements.

Wind direction provides an important but not infallible source of information on the direction of potential emissions sources. For example, when the wind direction is changing frequently, a measured concentration may also be from an emitted plume that has been blown back to the source. Large obstructions such as tanks also have wakes that can generate local winds opposite of the prevailing wind direction. Additionally, the AirMar (wind speed and direction sensor) is located on top of the moving vehicle and can be affected by the vehicle slipstream at higher speeds. To avoid issues with vehicle slipstream causing erroneous wind data, the data is only recorded when the vehicle's speed is less than 25 miles per hour. The wind direction is determined with an internal magnetic compass that also may be affected by local magnetic fields and large, nearby metallic objects.