ELSEVIER

**Fuel
Processing
Technology**

Available online at www.sciencedirect.com

SciVerse ScienceDirect

Simple and sensitive method for the measurement of volatile alkyl mercaptans in gasoline for remote field deployment

Siranoush Shahrzad [a,*], James Ford [a], Cheryl Sagara [a], Martin Flatley [b]

[a] Investigative Science Inc., 1050 Cooke Blvd., Unit #2, Burlington, Ontario L7T 4A8, Canada
[b] Suncor Energy Inc., Stn Main, Sarnia, Ontario N7T 7J3, Canada

ABSTRACT

Trace sulfur species, such as elemental sulfur, hydrogen sulfide, and volatile alkyl mercaptans, adversely affect silver based devices, such as gasoline level sensors, used in most vehicles. Identification and measurement of the sulfur species are key to developing a process control and/or treatment system to solve the problem at the refinery, prior to product distribution. A new and innovative method was developed for the analysis of specific mercaptan species in gasoline, and is reported here. The initial sample treatment stage is precipitation and stabilization of mercaptans as silver mercaptides. This stabilization could be successfully utilized in the field to mitigate the instability of mercaptans in gasoline samples. Another advantage of this step is that silver nitrate (AgNO$_3$) precipitation concentrates the mercaptans allowing for trace level detection. The silver mercaptides are converted to pentafluorobenzyl derivatives and analyzed by gas chromatography (GC) with mass spectrometric (MS) detection. Results based on this technique show that alkyl mercaptans can be accurately measured in the full scan mode of MS with a limit of detection (LOD) of 0.02 mg/L when a 100 mL gasoline sample is used. The recovery of mercaptans spiked into gasoline was 85 to 120% (n = 5). Gasoline producers will find knowing the exact levels of mercaptans in their products a useful tool to avoid distribution delays.

© 2013 Elsevier B.V. All rights reserved.

1. Introduction

Incidents of vehicle fuel level sensor/sender failures have been reported worldwide since 1999 and thousands of motorists have been affected by it. Electrical contacts in fuel level sensors made of silver (Ag alloys) are vulnerable to corrosion by certain commonly occurring trace sulfur species found in gasoline. This corrosion can lead to inaccurate/false dash board fuel level readings. Silver-plated bearings used in some gas station fuel pumps are another example of components that may be adversely affected by the sulfur species in fuel.

The corrosion mechanisms are not fully understood but thought to be due to synergism between elemental sulfur, hydrogen sulfide, and specifically low molecular weight mercaptans [1,2]. Levels of contamination in the low ppm range may cause this problem — although the specifics are not entirely understood. Gasoline with this sulfur contamination cannot be distributed and sold, posing a logistical problem for refineries. Total sulfur measurements at the refinery are not useful in predicting mercaptan concentration or species in gasoline. The Petro Canada silver wool test [3] works well as a predictor of the likelihood of mercaptan containing gasoline to damage automobile sensors, but does not identify the specific sulfur species. Identification of the sulfur species (in this case mercaptan) is key to developing a process control and/or treatment system to solve the problem at the refinery level.

While there are several analytical methods that can be employed for the measurement of total mercaptans in gasoline [4–8], to the best of our knowledge, there are no reports on reliable measurement of specific mercaptans in gasoline at concentrations lower than 1 ppm. The short holding time of low molecular weight mercaptans in the samples is also one limitation of most available methods. Several studies have described analysis methods for sulfur components in gasoline using GC coupled with sulfur-specific detectors [9,10]. However, these methods only determine that sulfur-containing compounds are present in gasoline and do not specify any molecular structures or distinguish peaks corresponding to specific mercaptans. Derivatization of mercaptans for analysis by GC or HPLC was found to be a reliable approach for identification and quantification of mercaptans [11–13]. Measurement of mercaptans after derivatization with pentafluorobenzyl p-toluenesulfonate was reported for mercaptan standards dissolved in dichloromethane by Funazo et al. [11]. Wu et al. [12] used pentafluorobenzyl bromide (PFBBr) for derivatization and measured both n-butyl and phenyl mercaptan standards in acetone. These methods introduced the concept of derivatization, and used low volumes (no more than 0.5 mL) of pure standards as analyte. They did not suggest any methodology for measurement of entrained mercaptans (i.e. in gasoline). Nishikawa et al. [13] determined levels in air samples by derivatization of low molecular weight mercaptans with 7-chloro-4-nitro-2,1,3-benzoxadiazole and measured them by HPLC. These kinds of derivatizations and measurements by HPLC or GC would be useful for the development of methods for the determination of mercaptans in gasoline if we include extraction steps for

gasoline samples to achieve a detectable amount of mercaptans. To determine trace amounts of mercaptans in gasoline (in the range of 0.1 ppm), an extraction of at least 25 mL of gasoline is needed. In recent years the technology for determination of mercaptans in evaporated samples or gas (state) has been greatly improved. The specialized sampling filters [14] and/or sophisticated GC systems and GC columns [15,16] allowed identification and measurement of a large number of mercaptans in gas (state) samples without any derivatization and with detection limits as low as 0.2 ppm (v/v). Several GC based instruments, as sulfur compound or mercaptan analyzers, have become commercially available for analysis of ambient air (e.g.: TRSMEDOR — H2S, mercaptans' analyzers M52022 or the Syntech Spectras GC955 series 800). These methods all use large volumes of gas (state) samples and, therefore, are not useful or sensitive enough for analyzing liquid gasoline samples.

This report introduces a method for stabilizing and analyzing mercaptans in gasoline with consideration of three critical aspects of this kind of analysis:

I) The mercaptans in gasoline samples should be stabilized at the time of sampling as they may degrade or evaporate.
II) The mercaptans need to be concentrated in the samples to be at measurable levels.
III) A method for the analysis should be selected, which is highly sensitive, selective and deals with stable derivatives of the mercaptans.

A novel, simple, practical, easy-to-implement method was developed to quantify the specific mercaptan species in gasoline at low ppm (mg/L) levels for remote field deployment. The idea was adapted from Wu et al. [12], who analyzed standard n-butyl and phenyl mercaptans by silver precipitation and derivatization. The method [12] was modified by adding an extraction step for high volumes of gasoline, using mass spectrometric (MS) detection, which offers positive identification and sensitive quantification ability, and some other adjustments described in this paper. The method was thoroughly validated for work with gasoline samples from refineries. It was demonstrated that when mercaptans in gasoline are precipitated by silver nitrate, the precipitation step served to stabilize and concentrate the mercaptans in the precipitate. Two model mercaptans, ethyl and n-propyl mercaptans, were analyzed.

2. Materials and methods

2.1. Materials

Ethyl and n-propyl mercaptans were purchased from Sigma-Aldrich. Pentafluorobenzyl bromide (98%) was purchased from Alfa Aesar. Other reagents (e.g. n-hexanes, xylenes, and silver nitrate) were of analytical reagent grade. Mercaptan standard solutions were prepared in xylenes. n-Tetradecane-d$_{30}$ (purchased from C/D/N Isotopes Inc.) was used as an internal standard.

2.2. Preparation of Clean Gasoline

A regular grade gasoline sample was provided by Suncor Energy Inc. Any existing mercaptans were removed from the gasoline sample by two sequential manual extractions with 3 N NaOH [17] followed by three sequential manual extractions with pure water (to ensure no NaOH remained in the gasoline). This extracted sample was referred to as Clean Gasoline and was used as a control. Ethyl and n-propyl mercaptan standards were spiked in sub-samples of Clean Gasoline. These samples were treated and analyzed as per the method and their analytical results were used to calculate the recovery of mercaptans in gasoline. (Note: The mercaptan standards in gasoline were unstable. All spiked solutions were used within 6 h of preparation.)

2.3. Extraction and derivatization procedure

Ethyl and n-propyl mercaptans were precipitated as silver mercaptides, derivatized, and analyzed by GC/MS. For the calibration curve, an aliquot of the standard solution was added to 6 mL of 0.02 M AgNO$_3$ (dissolved in isopropanol) solution in a 15 mL glass test tube with a Teflon lined cap. After the test tube was shaken by hand to attain the complete formation of silver mercaptide precipitate, it was centrifuged at 670 g for 5 min. The supernatant was removed and 1 mL of deionized water was added to the precipitate. After shaking, 0.1 mL of Na$_2$S (0.1 M), 0.125 mL of ZnSO$_4$ (0.04 M) and 0.1 mL of NaOH (8 M) solutions were added in succession. Then, 2.0 mL of the internal standard solution (0.127 mM in n-hexane) and 10 μL of PFBBr were added. The reaction mixture was shaken for 1 h in a water incubator at 60 °C. An aliquot of 1 mL of the clear n-hexane layer was transferred to a 4 mL vial, dried by blowing nitrogen on it, and the precipitate was dissolved in 100 μL n-hexanes. One microliter of this solution was injected into the GC.

To analyze gasoline samples, 40 mL of gasoline was added to 6 mL of 0.02 M AgNO$_3$ (dissolved in isopropanol) solution, the mixture was centrifuged at 670 g for 5 min and the supernatant was removed. 1 mL deionized water was added to the final precipitate and it was treated as described above.

2.4. Gas chromatographic conditions

Both pentafluorobenzyl derivatives of ethyl and n-propyl mercaptans were analyzed by GC/MS. The GC/MS was comprised of a Hewlett Packard (HP) 5890 Series II GC and a HP 5970 Series Mass Selective detector. The column (length: 30 m, diameter: 0.250 mm) was a DB-5 ((5%-Phenyl)-methylpolysiloxane column; 300/320 °C temperature limit; 0.25 μm coating thickness; from Agilent J&W). The injector temperature was kept at 250 °C. The oven temperature program was 3 min isothermal at 50 °C and then a 10 °C/min ramp to 300 °C, held for 2 min. Helium was used as carrier gas. A split flow was used. The interface temperature was 250 °C and peak areas were measured by a computer using the program HP Chemstation. The MS was operated in full scan mode, screening from 50 to 400 amu with 1.7 scans per second. To improve the separation and detection, selected single ions (e.g. parent ions at $m/z = 242$ and $m/z = 256$, which correspond to pentafluorobenzyl ethyl thioether and pentafluorobenzyl n-propyl thioether, respectively, and $m/z = 82$ to correspond with the internal standard) were monitored and areas under those peaks were used for quantification.

3. Results and discussion

3.1. Analytical calibration

A calibration graph for ethyl and n-propyl mercaptans, plotting the peak area ratio of the derivative to the internal standard against the mercaptan weight is shown in Fig. 1. This graph covers the range of 3.5–280 μg (0.035–2.8 mg/L when 100 mL gasoline was analyzed). The linear regression equations, y = 0.0014x + 0.0038 with a correlation coefficient of 0.999 for ethyl mercaptan and y = 0.0007x + 0.0036 with a correlation coefficient of 0.990 for n-propyl mercaptan, were obtained. The results indicate linearity over the range analyzed. The observed limit of detection (LOD) was 1.7 μg. The LOD was also calculated from the results of seven independently prepared 13.9 μg mercaptans in Clean Gasoline standards by using the following formula [18].

$$LOD = t(n-1) \times SD.$$

Where t (n − 1) is the value of the 95% one-side Student's distribution for n − 1 degrees of freedom, and SD is the standard deviation obtained for seven replications. The LOD calculated for the mercaptan
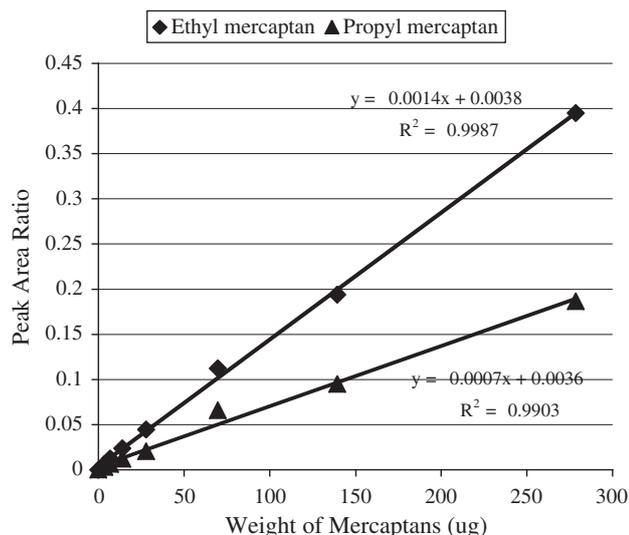
Fig. 1. Calibration graph for ethyl mercaptan (♦) and propyl mercaptan (▴).

Fig. 3. GC/mass ion plot chromatograms of the derivatives of ethyl and n-propyl mercaptans; the peaks (A), and (B) correspond to the derivatives of ethyl and n-propyl mercaptans, respectively; the peak (C) corresponds to the internal standard and peak (D) corresponds to the pentafluorobenzyl ion.

derivatives using seven replicates was 2.3 μg, which corresponds to the LOD of 0.023 mg/L (ppm) in gasoline (when 100 mL of gasoline was analyzed).

The typical full scan chromatogram of the derivatives of ethyl and n-propyl mercaptans spiked in gasoline as illustrated in Fig. 2 displays well resolved and highly symmetrical peaks. Fig. 3 displays ion plot chromatograms where peaks (A) and (B) correspond to the derivatives of ethyl and n-propyl mercaptans, respectively. Peak (C) corresponds to the internal standard and peak (D) corresponds to pentafluorobenzyl ion. The mass spectra of the derivatives are shown in Fig. 4. The mass spectra exhibit the parent ions at $m/z = 242$ and $m/z = 256$, which correspond to pentafluorobenzyl ethyl thioether and pentafluorobenzyl n-propyl thioether, respectively (Fig. 4(A) and (B)). The specific ion, $m/z = 181$, due to pentafluorobenzyl ion, of all the BFBBr derivatives was the most prominent ion in the mass spectrum. The molecular ions ($m/z = 242$ and $m/z = 256$) (Fig. 3) were used for specificity. For detection and quantification, higher sensitivity could be obtained using $m/z = 181$. All the chromatograms and the mass spectra in Figs. 3 and 4 were obtained by a single injection of 30 μg as mercaptans. Satisfactory peak shapes and separation were obtained for all concentrations and samples used in this paper.

It is possible to have 10 to 100 times greater sensitivity if the selected ion monitoring (SIM) mode is used, however, at the time of
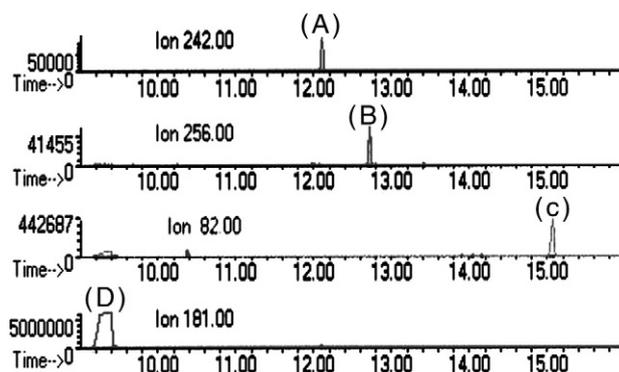
this method development a broader use of the method was desired, so open scan mode was used.

3.2. Reaction conditions

The reactions involved in this measurement were explained by Wu et al. [12] and are represented in equations as follows:

A) Mercaptans are precipitated as silver mercaptides

$$RS^- + Ag^+ \rightarrow RSAg\downarrow(\text{yellow precipitate}).$$

B) For a complete reaction between mercaptides and PFBBr the mercaptide ions should be liberated. Na$_2$S reacts with the excess amount of AgNO$_3$ and silver mercaptides to produce black Ag$_2$S precipitate and liberates mercaptide ions.

$$S^{2-} + 2Ag^+ \rightarrow Ag_2S\downarrow(\text{black precipitate})$$

$$S^{2-} + 2RSAg \rightarrow Ag_2S\downarrow + 2RS^-$$
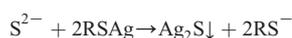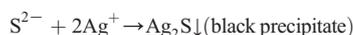
C) Free sulfide ions (excess from Step B) can also react with PFBBr, and are precipitated by the addition of ZnSO$_4$.

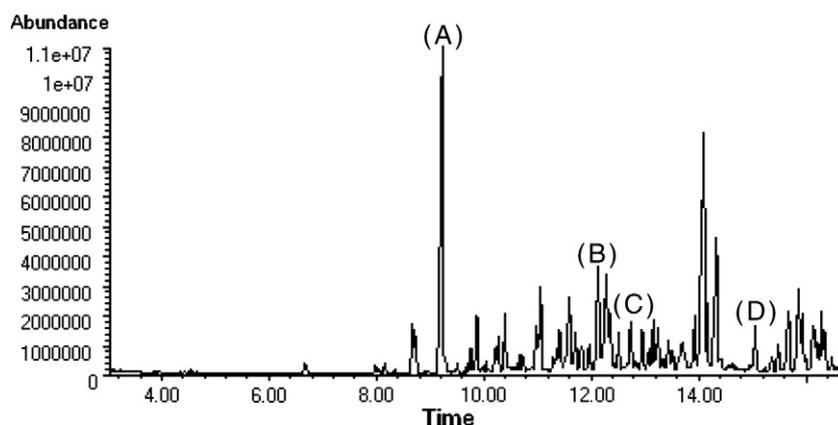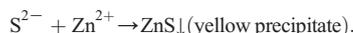$$S^{2-} + Zn^{2+} \rightarrow ZnS\downarrow(\text{yellow precipitate}).$$

Fig. 2. The typical full scan chromatogram of the derivatives of ethyl and n-propyl mercaptans spiked in gasoline. Peak (A) corresponds to the pentafluorobenzyl ion. Peaks (B) and (C) correspond to the derivatives of ethyl and n-propyl mercaptans and peak (D) corresponds to the internal standard, n-Tetradecane-d$_{30}$.

D) Mercaptide ions react with PFBBr in an alkaline medium with incubation at 60 °C for 1 h.
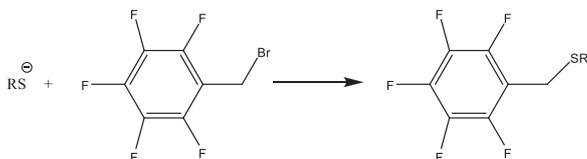
To optimize the PFBBr derivatization reaction for the mercaptans at the trace level, the yields of the derivatives were examined by changing the reaction time. The reaction time required to obtain a constant derivatization yield is approximately 1 h for both ethyl and n-propyl mercaptans. Therefore, the reaction time was fixed at 1 h for these mercaptans. The yields of the derivatives were the same using incubation temperatures of 60 °C or 70 °C. The 60 °C temperature was used for these experiments to minimize the loss of n-hexanes during the incubation time.

3.3. Recovery and detection of mercaptans in gasoline

The derivatization yield of each mercaptan from gasoline through silver mercaptide was similar to the yield when standard mercaptan was derivatized by the same procedure as described above. A recovery of 85–120% was obtained for ethyl and n-propyl mercaptans when they were spiked in Clean Gasoline (Table 1). The coefficient of variation was less than 11% (n = 5). Among seven different gasoline samples collected by Suncor Energy Inc. (real field samples not spiked), small levels of ethyl and propyl mercaptans were detected (Table 1). This method can effectively determine the specific mercaptan species and their concentrations in gasoline. The samples analyzed and reported here are proof of the method's effectiveness, however the concentrations found are not likely representative of freshly produced gasoline because the mercaptans are known to be volatile. Two Suncor Energy Inc. downstream fuel depots each had a sample collected and analyzed in duplicate less than an hour after release. These samples were specifically analyzed to demonstrate low concentrations of low molecular weight mercaptans. The samples contained less than 0.8 mg/L ethyl mercaptan plus propyl mercaptan. Despite the very low levels of mercaptans in these gasoline samples, sharp and symmetric peaks were obtained with acceptable values of the relative percent difference (RPD, %) for duplicate samples (Table 2). This demonstrates both the sensitivity and precision of the method using real field samples. These results were valuable for Suncor Energy Inc. because the Coordinating Research Council (CRC) Report No. 653 (Silver Fuel Level Sensor Corrosion Program) reported that "Results showed that elemental sulfur (S8) alone did not cause silver corrosion in laboratory tests. Interactions between elemental sulfur, hydrogen sulfide (H2S), and mercaptans (ethyl and propyl) were necessary to cause silver corrosion" [2]. However, in Canada, there are no regulatory levels for specific sulfur species. According to the Canadian Environmental Protection Act, 1999; SOR/99-236 [19], the national standard for gasoline quality specification only prescribes that total sulfur content should not exceed 40 mg/kg. The average total sulfur content measured at Suncor Energy Inc. was 25 mg/kg in year 2012.

3.4. Field method stability results

The ability of stabilizing the mercaptans in gasoline samples directly following production makes this method both practical and advantageous for use at remote field locations. In practice, the gasoline samples would be added to pre-prepared 250 mL amber bottles containing 6 mL of 0.02 M AgNO$_3$ in isopropanol. The samples would then be stable and could be kept at room temperature until analysis.
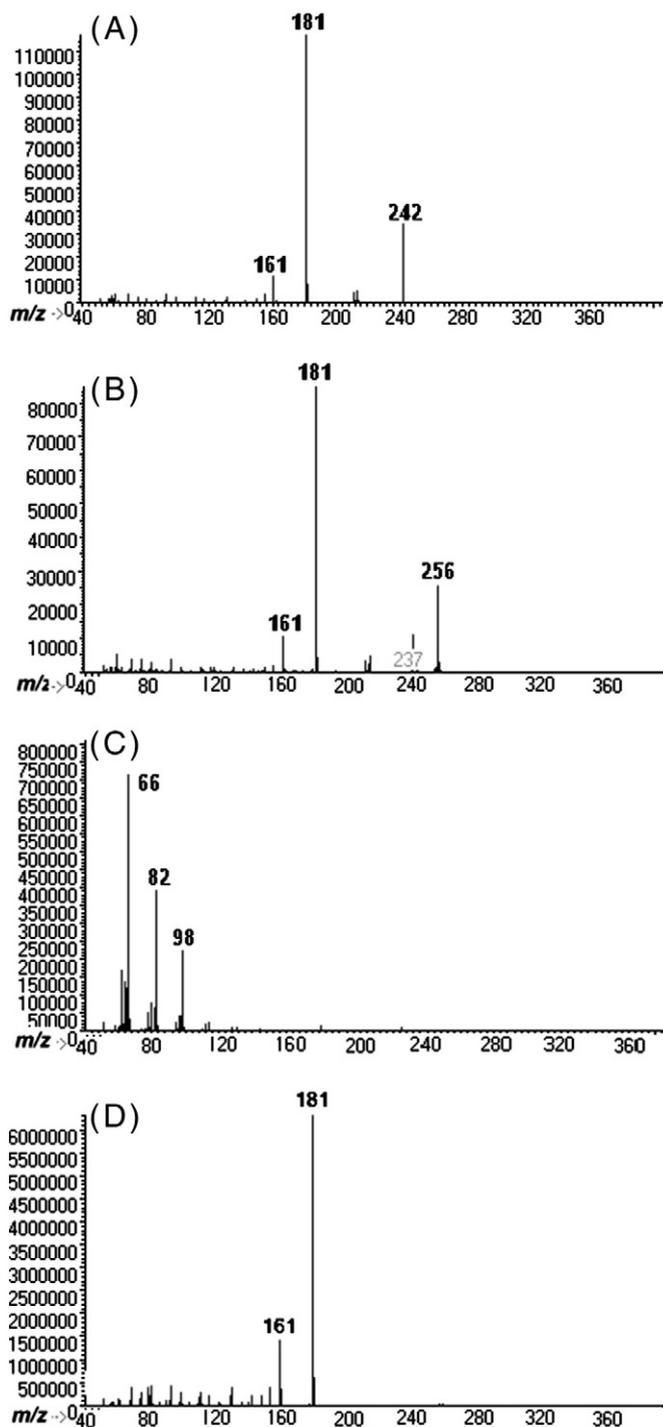

Fig. 4. Mass spectra of the peaks on the ion plot chromatograms of Fig. 2 (with the same sequence). (A) and (B) correspond to the parent ions of pentafluorobenzyl ethyl thioether and pentafluorobenzyl n-propyl thioether, respectively. (C) corresponds to the mass spectrum of the internal standard and (D) with the specific $m/z = 181$ corresponds to pentafluorobenzyl ion, which is also the most intensive ion of all the derivatives of PFBBr.

To determine the stability of mercaptide precipitates, 280 µg ethyl mercaptan and 210 µg propyl mercaptan were spiked in 40 mL Clean Gasoline. These samples were then treated as if they were field samples and placed in 250 mL amber bottles containing silver nitrate in isopropanol as listed above and analyzed at different time points; after 0 (same day analysis), 1, 2, 4, and 6 days. No significant difference was found over the course of these timepoints. The detailed results are given in Table 3.

Table 1
Measurement of the mercaptans in spiked (280 μg ethyl mercaptan and 210 μg propyl mercaptan in 40 mL Clean Gasoline) and in 40 mL industrial gasoline samples.

Sample	Found (recovery)	
	Ethyl mercaptan (μg)	Propyl mercaptan (μg)
Spiked #1	331.2 (118.29)[a]	185.2 (88.2)
Spiked #2	334.3 (119.4)	221.2 (105.3)
Spiked #3	307.0 (109.7)	194.2 (92.5)
Spiked #4	296.5 (105.9)	238.8 (113.7)
Spiked #5	287.6 (102.7)	195.8 (93.2)
Sample #1	0	0
Sample #2	0	0
Sample #3	44	44
Sample #4	160	432
Sample #5	<LOD	28
Sample #6	0	<LOD
Sample #7	0	0

[a] Recovery (%).

4. Conclusion

As described in the Introduction, although there are many analytical reports and sophisticated instruments available for measuring mercaptans, none of them reported species specific mercaptan measurements in gasoline samples. There was a lack of a reliable method specifically analyzing gasoline samples. This report, for the first time, introduces a method to quantify the specific mercaptan species in gasoline with great selectivity, sensitivity, and precision. This was achieved by modifying the method for acetone of Wu et al. [12] with the development of a method for extraction of mercaptans from large volume (up to 100 mL) gasoline samples. Additionally, this method introduces the use of GC/MS for analysis of PFBBr mercaptan derivatives, which is superior to the reported gas chromatography with electron capture detector (GC/ECD) method [12].

Specifically this method has the following advantages:

The method is simple, sensitive, and analytically specific. GC/MS analysis is both qualitative and quantitative so the mercaptan species are both quantified and positively identified. Although ethyl and propyl mercaptans have been used as model mercaptans in this study, the chromatograms and the full scan of mass detection of samples contain the analytical information for many other mercaptans present in the samples. Using the specific standard curves and extraction of the molec-

Table 2
Down stream fuel depot sample analysis.

Sample from	Found (RPD[a])	
	Ethyl mercaptan (mg/L)	Propyl mercaptan (mg/L)
Depot #1	0.40 (4.6)	0.30 (32.4)
Depot #2	0.27 (13.5)	0.15 (3.3)

[a] Relative percent difference for duplicate samples (%) (RPD).

Table 3
Field method stability results in spiked gasoline samples.

(40 mL Clean Gasoline containing 280 μg ethyl mercaptan and 210 μg propyl mercaptan was added to 6 mL AgNO$_3$ (0.02 M) and analyzed).

Analysis after	Found (recovery)	
	Ethyl mercaptan (μg)	Propyl mercaptan (μg)
2 h	293.8 (113.7)[a]	220.9 (105.2)
1 day	318.3 (118.3)	223.2 (106.3)
2 days	331.2 (115.2)	185.2 (88.2)
4 days	322.6 (103.8)	261.1 (124.3)
6 days	290.6 (119.4)	224.3 (106.8)

[a] Recovery (%).

ular ions of specific target mercaptans in the chromatograms, the same sample derivatization and analysis could be used for measurement of other mercaptans.

This method gives the option of stabilizing and concentrating the mercaptans in the gasoline samples directly after production. Since ethyl and propyl mercaptans are inherently unstable, and generally in low levels in gasoline, this will be of direct interest to gasoline producers as it can be used for remote field deployment and reducing or avoiding distribution delays.

Acknowledgment

References

[1] C. Cole, Various sulfur species play role in making gasoline corrosive to silver fuel sensors, Octane Week, 2006. XXI , (n/a).
[2] Coordinating Research Council Inc., CRC report no. 653, CRC Performance Committee, Silver Fuel Level Sensor Corrosion Program, Alpharetta, GA, May 2009.
[3] S. McDermid, R. Chin, D.P. Surette, Determination of the corrosiveness to silver of gasoline and distillate fuels using silver wool rapid ultrasonic method, Petro-Canada PCM 1005-03-IV method, in: D.P. Surette (Ed.), Petro Canada Methods and Procedures fourth ed., 2005, (Revision 2.00, PC-QMD-1007-50).
[4] W. Lei, P.K. Dasgupta, S.D. Matza, D.C. Olson, Measurement of mercaptans in gasoline, Microchimica Acta 99 (1989) 35–41.
[5] X. Chenga, L. Huang, X. Fu, P. Li, Z. Hu, L. Cao, Investigation of the components in RFCC gasoline affecting the accuracy of potentiometric titration by GC–MS and GC–IR, Fuel Processing Technology 85 (2004) 379–390.
[6] ASTM D4952-09, Standard Test Method for Qualitative Analysis for Active Sulfur Species in Fuels and Solvents (Doctor Test), 2009.
[7] ASTM D3227-04a, Standard Test Method for (Thiol Mercaptan) Sulfur in Gasoline, Kerosine, Aviation Turbine, and Distillate Fuels (Potentiometric Method), 2010.
[8] ASTM D 5453-00, Standard Test Method for Determination of Total Sulfur in Light Hydrocarbons, Motor Fuels and Oils by Ultraviolet Fluorescence, 2002.
[9] A. Stumpf, K. Tolvaj, M. Juhasz, Detailed analysis of sulfur compounds in gasoline range petroleum products with high-resolution gas chromatography–atomic emission detection using group-selective chemical treatment, Journal of Chromatography. A 819 (1998) 67–74.
[10] F.P. Di Sanzo, W. Bray, B. Chawla, Determination of the sulfur components of gasoline streams by capillary column gas chromatography with sulfur chemiluminescence detection, Journal of High Resolution Chromatography 17 (1994) 255–258.
[11] K. Funazo, M. Tanaka, T. Shono, Derivatization of mercaptans with pentafluorobenzyl p-toluenesulfonate for gas chromatographic determination, Analytical Sciences 3 (1987) 257–259.
[12] H.-L. Wu, K. Funazo, M. Tanaka, T. Shono, Derivatization-gas chromatographic determination of mercaptans, Analytical Letters 14 (1981) 1625–1635.
[13] Y. Nishikawa, K. Kuwata, Liquid chromatographic determination of low molecular weight alkylthiols in air via derivatization with 7-chloro-4-nitro-2, 1,3-benzoxadiazole, Analytical Chemistry 57 (1985) 1864–1868.
[14] NIOSH Test Method 2542 (Mercaptan, Methyl-, Ethyl-, n-Butyl), NIOSH Manual of Analytical Methods (NMAM), Fourth Edition, 1994.
[15] R. Gras, J. Luong, V. Carter, L. Sieben, H. Cortes, Practical method for the measurement of alkyl mercaptans in natural gas by multi-dimensional gas chromatography, capillary flow technology, and flame ionization detection, Journal of Chromatography. A 1216 (2009) 2776–2782.
[16] J. Ellis, A.K. Vickers, C. George, Capillary column selectivity and inertness for sulfur gas analysis in light hydrocarbon streams by gas chromatography, Fuel Chemistry Division Preprints 47 (2002) 703–704.
[17] D.L. Yabroff, Extraction of mercaptans with alkaline solutions, Industrial and Engineering Chemistry 32 (1940) 257–262.
[18] Environment Canada Manual of Analytical Methods, vol. 2, National Laboratory for Environmental Testing, Canadian Centre for Inland Waters, Burlington, Ontario, Canada, 1998.
[19] Canadian Environmental Protection Act, SOR/99-236, Sulphur in Gasoline Regulations, http://laws-lois.justice.gc.ca/PDF/SOR-99-236.pdf.