

Agilent AGM 1 Oxygen Gas Accessory and Organics Kit

User's Guide



Agilent Technologies

Notices

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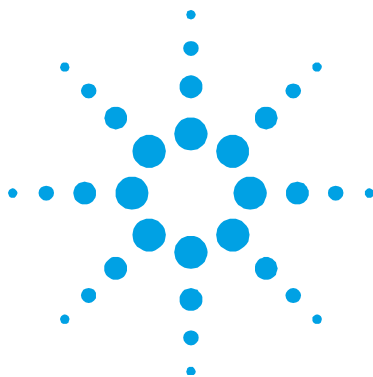
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Contents

1. Introduction	5
2. Installation	7
AGM 1 Oxygen Accessory	7
Gas requirements	8
Installing the AGM 1	8
Organics Kit	9
Installing the Organics Kit	10
Spraychamber	11
Pump Tubing and Nebulizer	12
3. Operation	15
The AGM Unit	15
Operating Procedure	16
AGM 1 Flow Calibration	17
4. Application Example	19
Determination of lead in unleaded gasoline by ICP-AES with the use of oxygen and a cooled spraychamber	20
Introduction	20
Experimental	21
Instrumentation	21
Standard Preparation	23
Sample Preparation	24
Results and Discussion	24

Effect of oxygen on Pb emission intensity	24
Effect of the addition of iodine and Aliquat 336 on Pb emission intensity	25
Selection of Pb wavelength	25
Washout time of the cooled spraychamber	27
Long term stability	27
Results	28
Conclusion	29
References	29
5. Spare Parts and Consumables	31



1. Introduction

The AGM 1 Auxiliary Gas Module is an accessory used to introduce a small amount of oxygen into the plasma of the ICP-OES spectrometer.

The oxygen facilitates the analysis of volatile organics by stabilizing the plasma when aspirating high vapor pressure organic solutions.

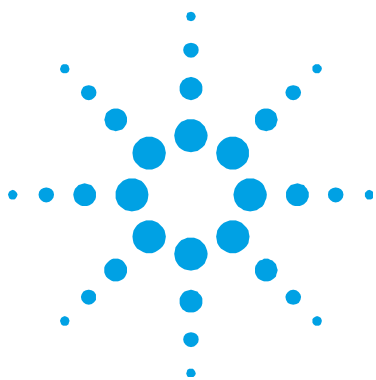
The AGM 1 Auxiliary Gas Module is generally used in conjunction with the Cooled Spraychamber. This allows you to analyze directly (ie, without dilution) organic solutions with a high vapor pressure at room temperature (eg, gasoline, naphtha). Cooling of the sample prevents high vapor pressures being generated in the spraychamber which can cause back pressure in the chamber and make the signal disappear.

The Cooled Spraychamber requires, but does not include, a cooler capable of cooling to approximately $-10\text{ }^{\circ}\text{C}$, and tubing to connect the cooler to the spraychamber.

This manual describes how to fit and operate the AGM 1 and the Cooled Spraychamber.

Introduction

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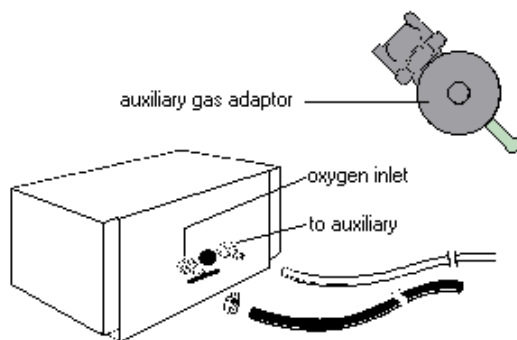


2. Installation

AGM 1 Oxygen Accessory	7
Gas requirements	8
Installing the AGM 1	8
Organics Kit	9
Installing the Organics Kit	10
Spraychamber	11
Pump Tubing and Nebulizer	12

AGM 1 Oxygen Accessory

Unpack the AGM 1. You should receive the following components.



You will need:

- 1 flat blade screw driver
- 1 brass standard barbed-tail gas hose connector to suit your
- 1 oxygen gas outlet

Gas Requirements

All gas supply installations must comply with rules and/or regulations that are imposed by the local authorities responsible for the use of compressed gas in the workplace.

Permissible pressure range: 300-700 Kpa (43 - 101 PSI)

Recommended pressure: 300 kPa (43 PSI)

Required flow rates: Up to 0.6 liters per minute

Installing the AGM 1

To install the AGM 1, follow the steps below:

- 1** Place the AGM 1 unit on a bench with the back facing towards you.
- 2** Take the Nalgene tubing (1/8 in. ID x 1/4 in. OD) supplied and fit it over the barb on the back of the AGM 1 unit marked 'To auxiliary'.
- 3** Slide the hose clamp onto one end of the black air hose. Place the hose and clamp over the barb on the AGM 1 marked 'Oxygen inlet'. Tighten the hose clamp with a flat blade screw driver.
- 4** To the other end of the black air hose, fit a gas hose fitting suitable for your laboratory oxygen supply.
- 5** Place the accessory in position between the laboratory oxygen supply and the sample introduction system.
- 6** Attach the black gas hose to your laboratory oxygen outlet (using a suitable connector). Do not turn on the gas supply at this stage.
- 7** Locate the auxiliary gas outlet in the sample compartment of the ICP-OES. If necessary, disconnect the torch auxiliary tubing from the outlet.
- 8** Fit the gas supply adaptor (supplied with the AGM 1) to the auxiliary gas outlet on the spectrometer. Fit the torch auxiliary tubing to the polypropylene end of the adaptor.
- 9** To the brass outlet of the adaptor, connect the free end of the Nalgene tubing fitted in Step 2 above.
- 10** The AGM 1 is now ready for use. Refer to Section 3 for operating instructions.

NOTE

The AGM 1 may be used on its own, but, to achieve the best results, it should be used with the cooled spraychamber.

Organics Kit

The cooled spraychamber comes in a kit with a demountable torch, tubing and attachments.

You will need to provide a cooler capable of cooling to approximately -10 0C and tubing to connect the cooler to the spraychamber.

In the Organics kit you should receive the following components:

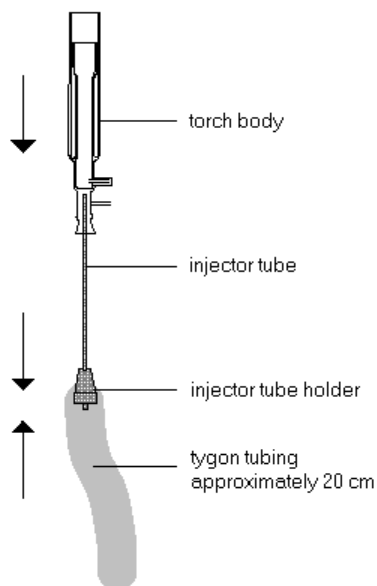
- Demountable torch body
- Demountable torch injector tube holder
- Small bore (0.8 mm ID) injector tube (pkt 3)
- Peristaltic pump tubings organics Black (sample)
- Peristaltic pump tubings organics Grey (drain)
- Transfer tubing for use with gasoline
- *Nebulizer holder
- *O-ring (for nebulizer)
- *O-ring kit (for nebulizer holder)
- Spraychamber tubing to drain
- Drain tubing
- Silicon tube
- Coupling insert, 2x
- Cooled spraychamber
- Mounting bracket
- Instruction sheet

*These items are assembled as one unit.

Installing the Organics Kit

To install the organics kit:

- 1 If the instrument is running switch off the plasma and allow the torch to cool.
- 2 Disconnect and remove the torch, spraychamber, nebulizer and pump tubing.
- 3 Assemble the components of the demountable torch as depicted in the figure below.



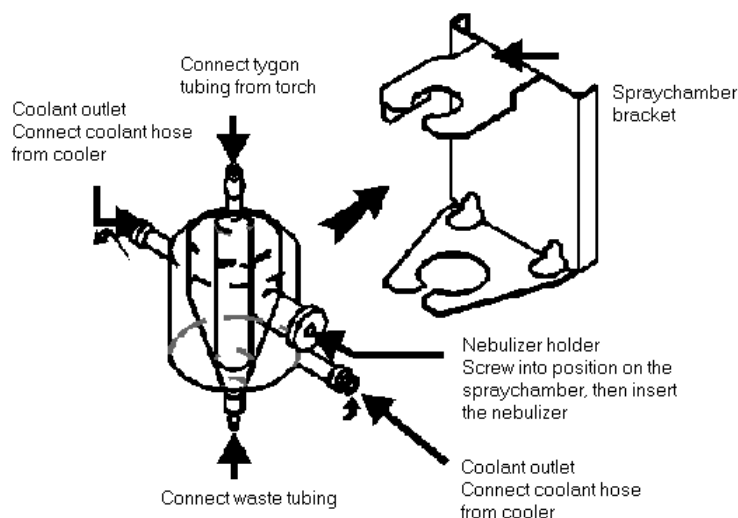
- 4 Fit the plasma and auxiliary gas hoses to the torch (refer to the instrument operation manual for further instructions).

NOTE

If you are using the cooled spraychamber kit with the AGM 1, attach the torch auxiliary gas hose to the brass end of the auxiliary gas adaptor (supplied with the AGM 1) fitted over the auxiliary gas outlet of the spectrometer.

- 5 Fit a length (approximately 20 cm) of the tygon tubing supplied to end of the torch. This will later be connected to the top of the spraychamber.
- 6 Install the torch as per the instructions in the operation manual supplied with your spectrometer or the ICP Expert II Help.

Spraychamber



To install the cooled spraychamber:

- 1 Screw the nebulizer holder onto the spraychamber.
- 2 Slide the spraychamber onto the bracket.
- 3 Place the spraychamber and bracket in position in the sample compartment. The bracket should sit over the three plastic knobs on the right side of the sample compartment.
- 4 Fit the tygon tubing attached to the bottom of the torch over the sample outlet at the top of the spraychamber.
- 5 Install the recirculating cooler as per the manufacturer's instructions.

NOTE

To operate the cooler down to $-10\text{ }^{\circ}\text{C}$, mix ethylene glycol (1:1) with the recirculating water to ensure the refrigerant does not freeze.

WARNING



Chemical Hazard

Ethylene glycol is highly corrosive and should be handled with care. Always wear appropriate safety clothing, gloves and eye protection when using ethylene glycol.

- 6 Connect the cooler hoses to the coolant inlet and outlet on the spraychamber (see figure previous page).

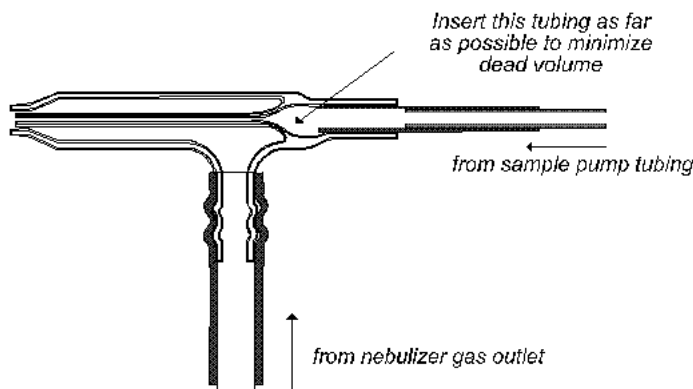
The drain tubing is attached to the outlet on the bottom of the spraychamber (refer to the 'Pump tubing and nebulizer' section below).

Pump Tubing and Nebulizer

A nebulizer is not provided with the cooled spraychamber kit. Use the 'K' style concentric glass nebulizer, supplied as standard with the instrument.

- 1 Select one piece of drain (Grey/Grey) pump tubing. Attach a connector barb (1/16 in. ID to 1/8 in. ID tube) to either end of the pump tubing.
- 2 Cut a length (approximately 30 cm) of the spraychamber to drain tubing (1.57 mm ID x 3.17 mm OD) provided. Attach it over the barb to one end of the drain pump tubing.
- 3 Cut a length of the drain tubing (1/8 in. ID x 3/16 in. OD) provided to reach between the peristaltic pump and your waste vessel. Attach it to the free end of the drain pump tubing.
- 4 Fit the drain pump tubing onto the peristaltic pump as described in the instrument operation manual. Connect the spraychamber to drain tubing to the bottom of the spraychamber. Place the drain tubing into the drain vessel.
- 5 Select one piece of sample (Black/Black) pump tubing.

- 6 Cut off the excess pieces of pump tubing (any piece of pump tubing extending over 1 cm past the tabs) to minimize dead space.
- 7 Take a small piece of excess sample pump tubing (approximately 2-3 cm in length) and insert it carefully into the sample end of the nebulizer. Cut a piece of polyethylene tubing (0.04 mm ID x 1.09 mm OD, cut to fit between the nebulizer and the peristaltic pump) and insert this as far as it will go into the pump tubing attached to the nebulizer (see figure below).



- 8 Insert the nebulizer into the nebulizer holder on the spraychamber.
- 9 Fit the other end of the polyethylene tubing into one end of the sample pump tubing.
- 10 To the other end of the sample pump tubing fit a length of capillary to reach the sample, or alternatively, connect it to the SPS-5 autosampler (refer to the SPS-5 Sample Preparation System operation manual for further instructions).
- 11 Connect the nebulizer gas hose (from the 'Nebulizer' gas outlet in the sample compartment of the ICP-OES) to the nebulizer gas inlet.

NOTE

Do not use the Argon Saturator Accessory (ASA) with the cooled spraychamber. Any water introduced into the spraychamber will ice up inside the spraychamber if operated below 0 °C.

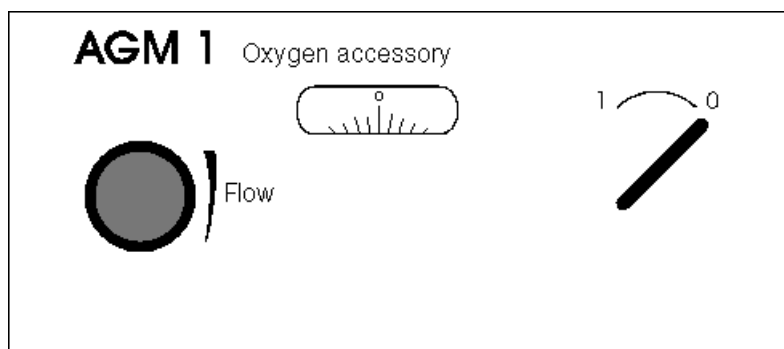
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3. Operation

The AGM Unit	15
Operating Procedure	16
AGM 1 Flow Calibration	17

The AGM Unit



The AGM 1 unit features an on/off switch, a flow control knob and a flow indicator.

On/Off switch

The on/off switch allows or prevents gas flow from the accessory to the instrument. This switch should always be set to '0' (off) until you are ready to use the accessory.

Flow knob and indicator

The flow knob controls the flow of oxygen to the instrument. The flow rate is displayed by the flow indicator.

Operating Procedure

NOTE

Do not use the ASA (Argon Saturator Accessory) with the Cooled Spraychamber. Water introduced into the spraychamber will ice up in the spraychamber if operated below 0 °C.

To operate your spectrometer with the organics kit installed:

- 1 Set all the accessory settings to off (gas flow to '0' and the On/Off switch to '0').
 - 2 Turn the laboratory oxygen supply on. Set the gas pressure to 300 kPa (the range is 300-700 kPa).
-

WARNING**Explosion Hazard**

A concentration of oxygen in the torch compartment may cause an explosion when the spark or plasma operates. Never turn on the oxygen unless both the exhaust and plasma are operating.

- 3 Aspirate an organic solution such as ethanol. Light the torch as per the instructions in the operation manual supplied with your spectrometer or the ICP Expert II Help.

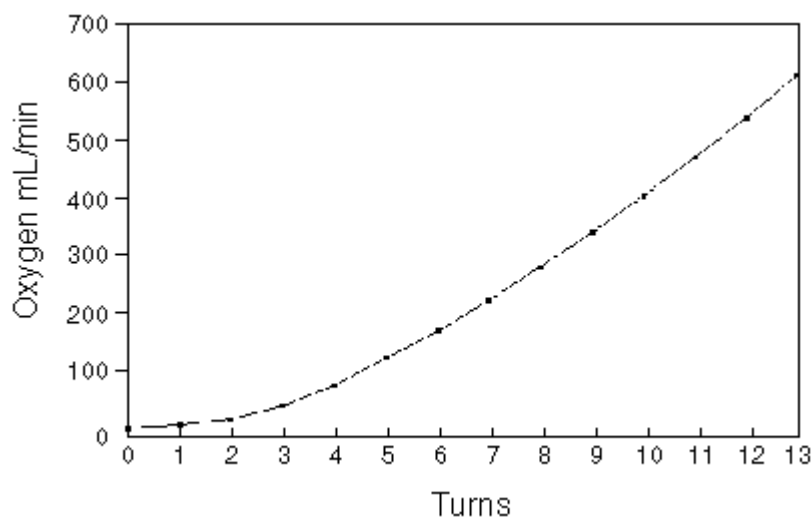
NOTE

When aspirating organic solutions, different torch lighting conditions may be required. If the torch fails to light using your standard torch lighting conditions, go to the Ignition Setup page and set the default settings. Then set the Run Power to 1.5 kW. Light the torch as usual.

- 4 Watch the plasma. The bullet and base of the plasma will appear green due to the excess carbon in the sample (C_2 band emission).
- 5 When you can see the green, turn on the AGM 1 gas flow by turning the switch to the '1' position.
- 6 Slowly turn the flow knob to supply oxygen to the instrument. Watch the plasma as the green disappears (approximately 3-4 turns on the flow indicator). This may take a while.

NOTE

The AGM 1 flow indicator is marked with arbitrary units. Use the calibration graph below to determine the actual gas flow.

AGM 1 Flow Calibration

The flow rate where the green color disappears from the plasma is the operating flow for the accessory.

- 7** Now introduce the sample. When the sample reaches the plasma, the green color will return to the plasma. This is normal.
- 8** Proceed with your method development, calibration and measurements in the normal way.

An analytical example using the AGM 1 and the cooled spraychamber is described in chapter 4 in this operation manual.

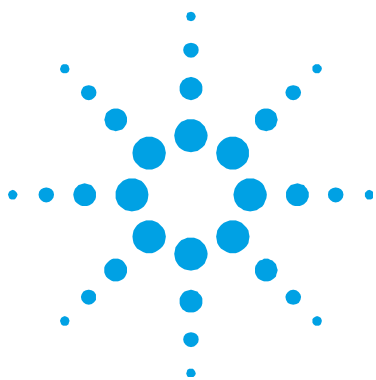
Operation

Below are some things to remember when using the AGM 1 and cooled spraychamber:

- ❑ Always set the pump speed (which controls the rate of sample delivery) to the lowest possible level. This will limit the flow of sample to the plasma and avoid the vapor pressure build-up in the spraychamber which may destabilize or extinguish the plasma. Never use fast pump with organic samples.
- ❑ Adjust the nebulizer gas flow so that the top of the bullet sits level with the top of the torch when aspirating sample. If the bullet sits too high or too low, the analytical signal may fluctuate.
- ❑ Bubbles may be seen in the sample stream (these generally occur once the solution has passed through the region being constrained by the pump tube holders). If the bubbles are causing your plasma to flicker, loosen the pump tube holding screws slightly.
- ❑ Always make up fresh standards to match the samples for analysis.

To turn off the AGM 1:

- 1 Slowly turn down the oxygen flow on the accessory. If you do this too quickly, the plasma may go out.
- 2 When the flow indicator reads '0', switch the on/off switch to '0'.



4. Application Example

Determination of lead in unleaded gasoline by ICP-AES with the use of oxygen and a cooled spraychamber	20
Introduction	20
Experimental	21
Instrumentation	21
Standard Preparation	23
Sample Preparation	24
Results and Discussion	24
Effect of oxygen on Pb emission intensity	24
Effect of the addition of iodine and Aliquat 336 on Pb emission intensity	25
Selection of Pb wavelength	25
Washout time of the cooled spraychamber	27
Long term stability	27
Results	28
Conclusion	29
References	29

The following application note can also be found on the Agilent Technologies website at www.agilent.com

NOTE

The Liberty is an older model ICP-OES and is also described as ICP-AES.

Determination of lead in unleaded gasoline by ICP-AES with the use of oxygen and a cooled spraychamber

No. 15

September 1993

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Varian Australia Pty Ltd

Mulgrave, Victoria, Australia

Introduction

In nature, lead is not highly abundant in the earth's crust. However, large amounts of lead have been released into the environment by human activities in the last few decades. The effects of lead on human health have been widely reported⁽¹⁻³⁾. The major contribution of lead into the atmosphere results from the combustion of gasoline. To improve the octane number, organo-lead compounds such as tetraethyl lead and/or tetramethyl lead have been used as anti-knock additives in gasoline. Since gasoline engines are the most common internal combustion engines for vehicles, the amount of lead released into the atmosphere from the exhaust gases of vehicles using leaded gasoline is high.

In 1986, the United States Environmental Protection Agency (USEPA) reported⁽⁴⁾ that 90% of the total lead released into the environment in the United States was the result of lead emission from gasoline combustion. In recent years, unleaded gasoline has been introduced in order to reduce lead pollution into the environment, and therefore, lead contamination in unleaded gasoline is closely monitored.

The determination of lead in gasoline has been described by various analytical techniques, such as X-Ray Fluorescence spectrometry (XRF)^(5,6), Colorimetry⁽⁷⁾, Volumetry⁽⁸⁾, Titrimetry^(9,10) and Atomic Absorption Spectrometry (AAS)⁽¹¹⁻¹³⁾. However, such analysis by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES) has not yet been described. ICP-AES is a technique that offers large linear dynamic range, relative freedom from chemical interferences, and multi-element capability.

Gasoline is a highly volatile liquid with a very low boiling point and extremely high vapor pressure, and its continuous aspiration into the plasma can lead to the rapid build up of carbon on the rim of the intermediate tube as well as the injector tip of the torch. The plasma can be rapidly extinguished.

To reduce carbon build up and maintain a stable plasma, oxygen is added and mixed with the auxiliary argon gas flow to assist the oxidation of carbon molecules. Unleaded gasoline can then be introduced into the plasma, and no more carbon build up is observed on the torch. On the other hand, the extreme high vapor pressure from the gasoline results in gradual accumulation inside the spraychamber with time, which causes the plasma to flicker and affects signal stability. By lowering the temperature of the gasoline (using a cooled spraychamber), the solvent vapor loading in the plasma is reduced: this results in a much more stable plasma.

This work describes the determination of Pb in unleaded gasoline by ICP-AES with the addition of oxygen and using a cooled spraychamber. The accuracy and validity of the method has been assessed by the use of a National Institute of Standards and Technology (NIST) Standard Reference Material (SRM) 2712 Pb in Reference Fuel, which contains tetraethyl lead.

Experimental

Instrumentation

A Varian Liberty 220 Inductively Coupled Plasma Atomic Emission Spectrometer was used for all the measurements. The resolution of the spectrometer is typically 0.018 nm in 1st order, 0.009 nm in 2nd order, 0.007 nm in 3rd order and 0.006 nm in 4th order. The operating conditions used are listed in Table 1.

An oxygen accessory AGM 1 (Varian Australia Pty. Ltd., Mulgrave, Australia) was used to introduce oxygen into the auxiliary argon gas flow before entering the torch, with its flow controlled by a high precision needle valve and the oxygen settings were displayed on the front panel of the accessory.

Application Example

Table 1. ICP operating parameters

Power	1.5 kW
Plasma gas flow	15.0 L/min
Auxiliary gas flow	2.25 L/min
Torch type	Demountable torch with a 0.8 mm I.D. injector tube
Nebulizer type	Concentric glass type K
Nebulizer pressure	80 kPa
Sample uptake rate	0.2 mL/min
Integration time	5 s
Viewing height	8 mm
Background correction	Dynamic
Grating order	2nd
Filter	Default
PMT voltage	650 V

A cooled glass spraychamber (Varian Australia Pty Ltd., Mulgrave, Victoria, Australia) and a glass concentric nebulizer were used. The cooled spraychamber was operated at a temperature of $-10\text{ }^{\circ}\text{C}$ with 1:1 ethylene glycol used as coolant and controlled by a refrigerated circulator. A schematic diagram of the cooled spraychamber is shown in Figure 1.

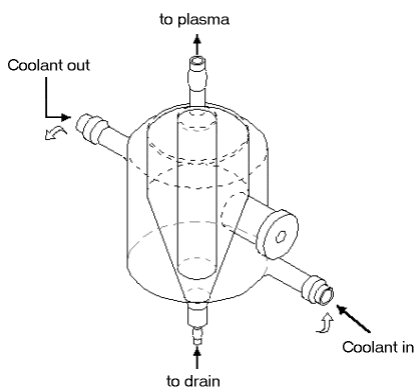


Figure 1. Schematic diagram of the cooled spraychamber

A demountable torch with a 0.8 mm I.D. injector tube was used. A small bore injector can give better tolerance to volatile organics compared to the 1.4 mm I.D. of a standard torch.

Analytical reagent grade organic solvents and chemicals were used.

- Iso-octane, Merck
- Toluene, BDH
- Dekalin (decahydronaphthalene), BDH
- Aliquat 336 (tricaprylmethylammonium chloride), Aldrich Chemicals
- Iodine, Univar, Ajax Chemicals
- 10% v/v Aliquat 336/dekalin solution: dissolve 10 mL (or 8.8 g) of Aliquat 336 into 100 mL of dekalin
- 3% w/v Iodine solution: dissolve 3 g of iodine crystals in toluene and dilute to 100 mL volume
- The unleaded gasoline sample was obtained from a service station

Dekalin was also used as the rinse solution

Standard Preparation

Stock standard was a 5000 mg/Kg Conostan Pb standard in hydrocarbon oil (Conostan division, Continental Oil Company, Ponca City, Oklahoma, USA).

100 mg/L Pb secondary standard solution was prepared by accurately weighing 2 g of the 5000 mg/Kg Pb standard into 100 mL of iso-octane.

Working standards were prepared by pipetting 1.25 mL and 2.50 mL of the 100 mg/L Pb standard solution into 25 mL volumetric flasks, then 20 mL of the unleaded gasoline sample was added to each flask, followed immediately by the addition of 0.3 mL of 3% iodine solution. This was allowed to stand for 5 minutes and mixed well. Then 0.5 mL of the 10% Aliquat 336/dekalin solution was added. It was made up to volume with dekalin and mixed well. The final solutions contained 5 mg/L Pb addition and 10 mg/L Pb addition, respectively.

Sample Preparation

20 mL of the unleaded gasoline sample was transferred to a 25 mL volumetric flask. 0.3 mL of 3% iodine solution was immediately added and allowed to react for 5 minutes. Then 0.5 mL of 10% Aliquat 336/dekalin solution was added, the solution was made up to volume with dekalin then mixed well.

For the NIST 2712 sample, 3 mL of the sample was transferred to a 25 mL volumetric flask, 20 mL of the unleaded gasoline sample was added, immediately followed by 0.3 mL of 3% iodine. This was allowed to stand for 5 minutes. Then 0.5 mL of 10% Aliquat 336/dekalin solution was added, made up to volume with dekalin, and mixed well.

Results and Discussion

The use of oxygen to improve the tolerance of the plasma to organic solvents has been reported⁽¹⁴⁻¹⁶⁾. In this work, oxygen was mixed with the auxiliary Ar gas flow. Carbon tends to form a deposit on the intermediate tube as well as the injector tip of the torch. The use of a high auxiliary flow of 2.25 L/min lifts the plasma up and when combined with the added oxygen prevents the build up of carbon. A stable plasma is achieved and, in addition, no safety hazard is presented by oxygen introduction in this manner. The use of a cooled spraychamber can reduce the solvent vapor loading in the plasma, resulting in a much more stable plasma for long term measurements on a routine basis.

Effect of oxygen on Pb emission intensity

It is essential to add oxygen to the argon gas flow to minimize carbon build up and sustain the plasma while gasoline is aspirated. In the experiment, oxygen was added before the gasoline was introduced in order to prevent the plasma from being extinguished. The amount of oxygen added to the auxiliary Ar flow to prevent carbon deposition on the torch, and provide optimum Pb emission intensity was studied. As can be seen in Figure 2, an oxygen flow rate of 0.035 L/min was found to be the optimum.

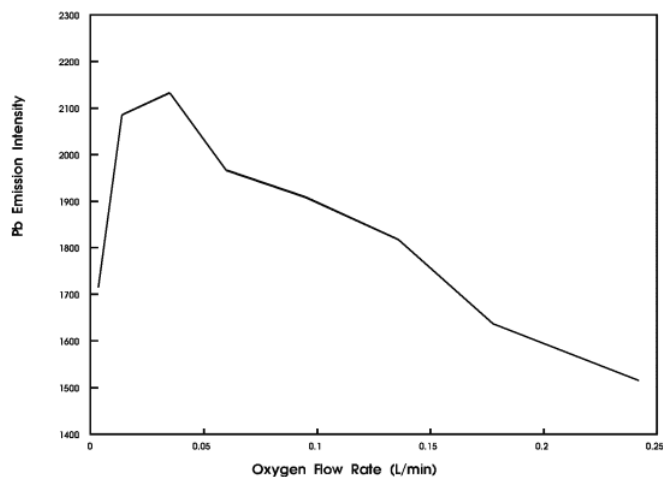


Figure 2. Effect of oxygen flow rate on Pb emission intensity

Effect of the addition of iodine and Aliquat 336 on Pb emission intensity

Experiments have shown that when iodine and Aliquat 336 were added, both the tetraethyl lead (from NIST 2712) and Conostan Pb standard give similar emission intensity at the same concentration. However, without the addition of those reagents, an apparent decrease in Pb signal was obtained with the NIST 2712 sample.

This is apparently because the alkyl lead compounds react with iodine to form iodo lead alkyl anions which then are stabilized by the addition of a quaternary ammonium salt such as Aliquat 336^(7,11,13). The stabilized iodo lead alkyl compounds are then analyzed by ICP.

Selection of Pb wavelength

The important criteria for the Pb wavelength selection is based upon the best detection limit and relative freedom from spectral interferences.

The most intense Pb line is 220.353 nm. However, at this Pb wavelength, a severe structural background is present when running organics. Therefore, other Pb lines such as those at 283.306 nm and the Pb 261.418 nm line were studied.

Application Example

The Pb 283.306 nm line has a detection limit of 0.050 mg/L and a background equivalent concentration of 5.3 mg/L, whereas the

Pb 261.418 nm line has a detection limit of 0.075 mg/L and a background equivalent concentration of 4.1 mg/L. The Pb 283 line was preferred because of its better detection limit and the closeness of the background equivalent concentration to the Pb 261 line.

Similar analytical results were obtained on both wavelengths.

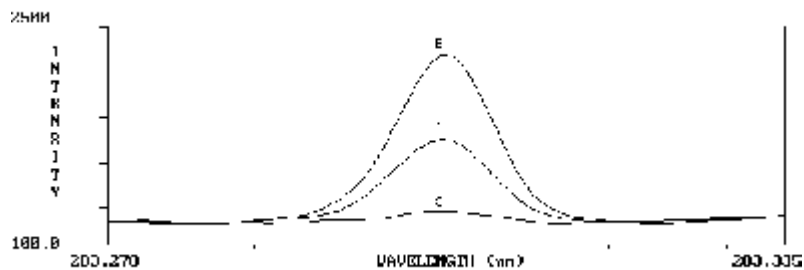


Figure 3. Spectral scans of (A) 5 mg/L Pb addition standard, (B) 10 mg/L Pb addition standard, and (C) unleaded gasoline sample at the Pb 283.306 nm line

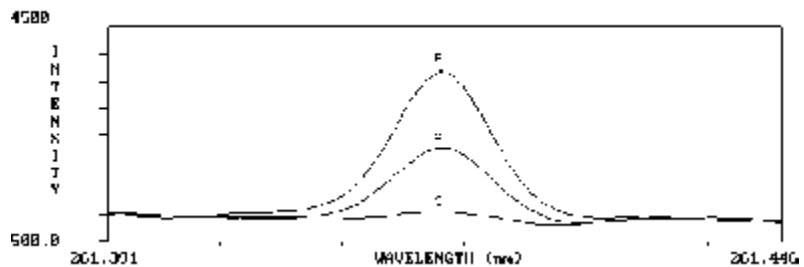


Figure 4. Spectral scans of (A) 5 mg/L Pb addition standard, (B) 10 mg/L Pb addition standards, and (C) unleaded gasoline sample at the Pb 261.418 nm line

Figures 3 and 4 show wavelength scans of 5 mg/L and 10 mg/L of Pb addition standards, and the unleaded gasoline sample in the vicinity of the Pb 283.306 nm line and Pb 261.418 nm line, respectively.

Washout time of the cooled spraychamber

The washout time of the cooled spraychamber was measured by aspirating a 1000 mg/L Pb in iso-octane for 5 minutes, then rinsing out with iso-octane while monitoring the signal decay of the

Pb 283.306 nm line signal intensity. The washout profile is shown in Figure 5. The time required for 1% decay of signal is 33 s, for 0.1% decay is 80 s and decay to background is 130 s. This was achieved with a sample uptake rate of 0.2 mL/min (refer Table 1).

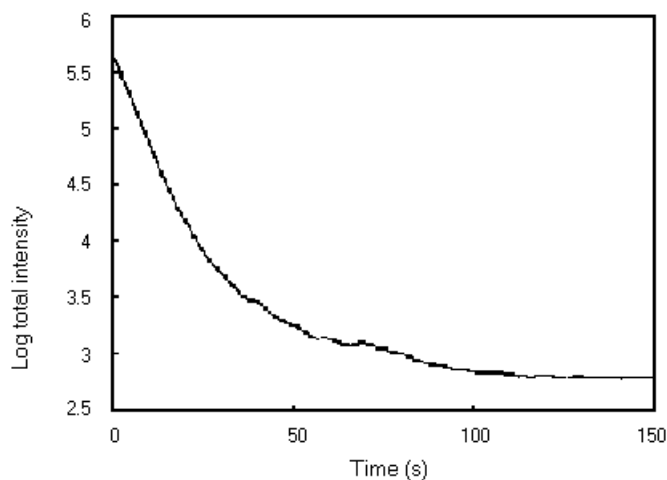


Figure 5. Washout profile of 1000 mg/L Pb in iso-octane by the cooled spraychamber at -10 °C

Long term stability

Long term stability was measured by continuously aspirating a 5 mg/L multi-element standard solution in unleaded gasoline for over an hour. As shown in Figure 6, the reproducibility of the measurements after an hour ranged from 0.7 to 1.1 %RSD.

Application Example

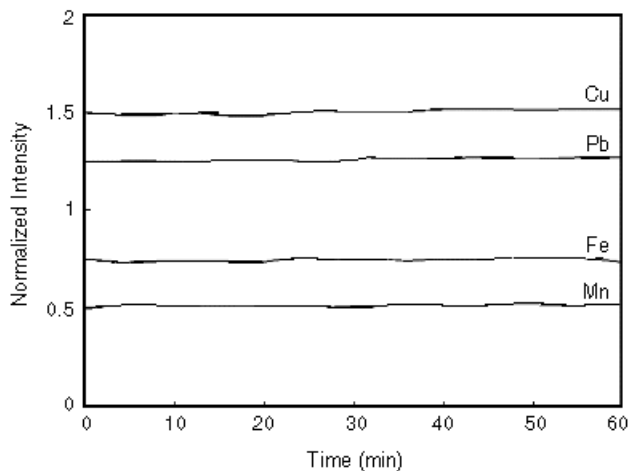


Figure 6. Signal stability over an hour for 5 mg/L of Mn, Fe, Pb and Cu spiked in unleaded gasoline sample.

Results

The standard addition method was used for calibration and the instrument operating conditions are listed in Table 1. The precision of the measurement ranged from 1 to 5 %RSD. The mean results of the triplicate analysis are listed in Table 2. The found value of the NIST SRM 2712 compares well with the certified value confirming the validity of the method and the accuracy of the measurements.

Table 2. Results of the analysis

Sample	Pb concentration (g/L)	
	Certified value	Measured value
NIST SRM 2712	0.0079 ± 0.0003	0.0080 ± 0.0002
Unleaded gasoline	-	0.00039 ± 0.00001

Conclusion

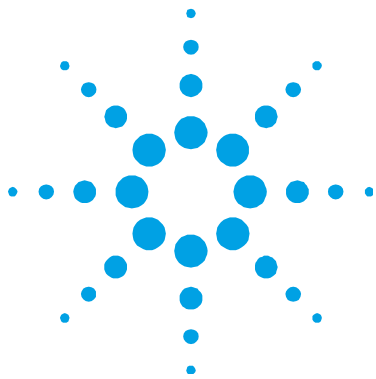
The determination of Pb in unleaded gasoline by ICP-AES with the use of oxygen and a cooled glass spraychamber has been described. The measured Pb value in NIST SRM 2712 is in good agreement with the certified value. The precision of the measurement ranged from 1 to 5 %RSD. The long term stability, which ranged from 0.7 to 1.1 %RSD, is excellent. This study has shown that ICP-AES is a suitable technique for the determination of Pb in unleaded gasoline.

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Application Example

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5. Spare Parts and Consumables

For ordering information, see the Agilent website at www.agilent.com.

The following spare parts are available from Agilent for the AGM 1:

- Nalgene tubing
- Rubber tubing
- Hose clamp

The following consumables and spares are available for the organics kit:

Cooled spraychamber

Peristaltic pump tubing organics Black (sample)

Peristaltic pump tubing organics Grey (drain)

Transfer tubing for use with gasoline

*Nebulizer holder

*O-ring (for nebulizer)

*O-ring kit (for nebulizer holder)

Drain tubing

Small bore (0.8 mm ID) injector tube (packet of 3)

Demountable torch body

Demountable torch injector tube holder

*The nebulizer holder consists of these three parts. You must reorder each part if you wish to replace the nebulizer holder.

Spare Parts and Consumables

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In This Book

The manual describes the following:

- Introduction
- Installation of the AGM 1 Oxygen Accessory and the Organics Kit
- Operating Procedures
- An Application Example
- Spare Parts and Consumables

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Issue 3



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