



## Multipoint calibration of an elemental analyzer with LPG standard and LPG's sample analysis

### Authors:

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This notice describes the calibration of a Multitek sulfur-nitrogen elemental analyzer and the analysis of sample with GasMix™ AIOLOS combined with AURA vaporizer in accordance with ASTM D6667 method.

### What is a LPG?

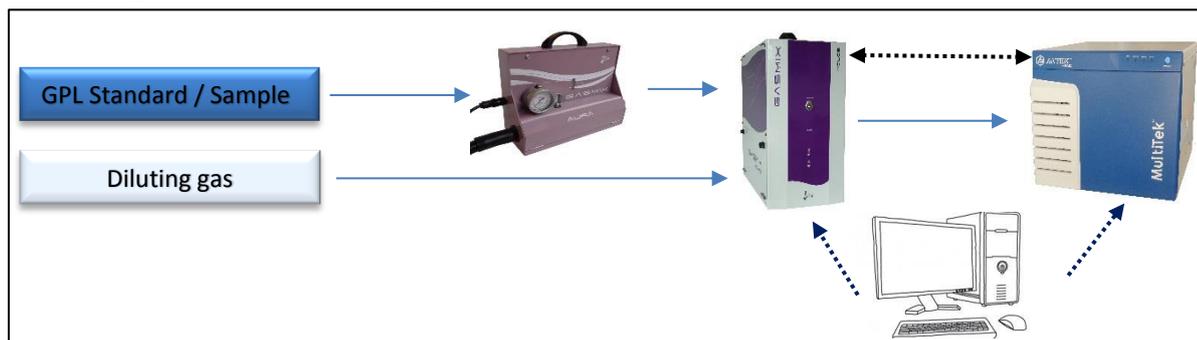
Liquefied petroleum gas (LPG) is a blend of light hydrocarbons derived from oil refining or natural gas treatment and stored in a liquid state. Propane and butane are predominant components. In normal temperature and pressure condition, they are in gaseous state, but for ease of transpiration, storage and retail LPG is liquefied.

Liquefied Petroleum Gas is considered as a « clean fuel », because in comparison to liquid fuels like gasoline and diesel, it has better combustion with reduced emissions of nitrogen oxide, carbon monoxide, hydrocarbons and particles. However, because it is used as fuel, it is necessary to control its sulfur and nitrogen content.

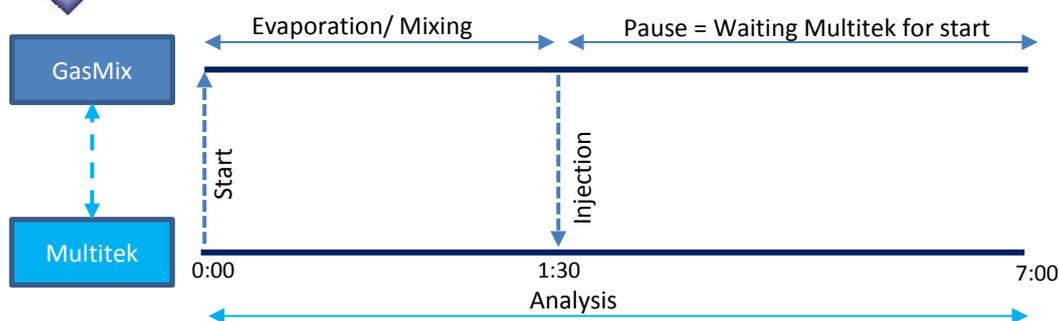
### Technical setup

The analyzer to calibrate is a Multitek from PAC. It measures the total sulfur in different matrices (gas, liquid or solid) by UV fluorescence. Our interest in this study was LPG that should be injected in the analyzer in gas phase. To assure proper and safe liquid-gas transformation, it was employed a vaporizer AURA developed by Alytech especially to analyze LPG and other liquefied gases. To set optimal evaporation temperature the AURA is equipped with a heated evaporator with regulator. It has heated transfer lines to prevent any condensation and build-in pressure regulator that keeps the output pressure at a desired value. In this study output pressure was set to 1 bar; evaporator temperature to 100°C and heated lines to 120°C. Thus, the injection system comprises an AURA vaporizer and GasMix™ AIOLOS II diluter equipped with an injection valve with a 10mL loop. Easy to use, with its associated software, GasMix diluter allows to create fully automated calibration sequence and calculates automatically the flows required to achieve the wanted concentrations.

A LPG standard containing about 100 ppm sulfur compound (dimethyl sulfide) was used for calibration. The diluent gas used is methane. A sequence of six different concentrations : 60, 45, 30, 15, 5.2, 2ppm and one blank was programmed with three replicas on each concentration.



**Figure 1:** Schematics of the installation



**Figure 2:** Automated calibration sequence

	Sequence GasMix
Number of cycle	3 cycles
Total time	3 min
Mixing time	2 min
Pause	1 min
Command relay	Close 1m58 s Open 2m20 s

**Table 1:** Details for the injection of one calibration point

With GasMix's software, all calibration points are done in a single completely automated sequence. The operator must only be present to connect the LPG cylinder and start the sequence.

The GasMix™ automatic sequence is started by the Multitek. It activates the injection valve and triggers the flow shutdown once the sample is injected, during analysis. Before each injection gas sample was sent to the vent for 1.5 min to purge all the lines including the injection loop and to ensure that any adsorption are stabilized. At the end of the cycle, GasMix™ is on stand by and wait for a next Multitek start signal.

Cycle	Nbr. cycles	Total	Voie 1	Voie 2	Voie 3	Voie 4	Total	Analyte sélectionné
# 1/7	3	00:09:00	0,0	55,0	0,0	...	55,0	...
# 2/7	3	00:09:00	1,1	53,9	0,0	...	55,0	DMS : 2,000 +/- 0,171
# 3/7	3	00:09:00	2,7	52,3	0,0	...	55,0	DMS : 5,000 +/- 0,196
# 4/7	3	00:09:00	8,1	46,9	0,0	...	55,0	DMS : 15,000 +/- 0,336
# 5/7	3	00:09:00	16,2	38,8	0,0	...	55,0	DMS : 30,000 +/- 0,605
# 6/7	3	00:09:00	24,3	30,7	0,0	...	55,0	DMS : 45,000 +/- 0,891
# 7/7	3	00:09:00	32,4	22,6	0,0	...	55,0	DMS : 60,000 +/- 1,182

**Figure 3 :** Calibration sequence on GasMix software

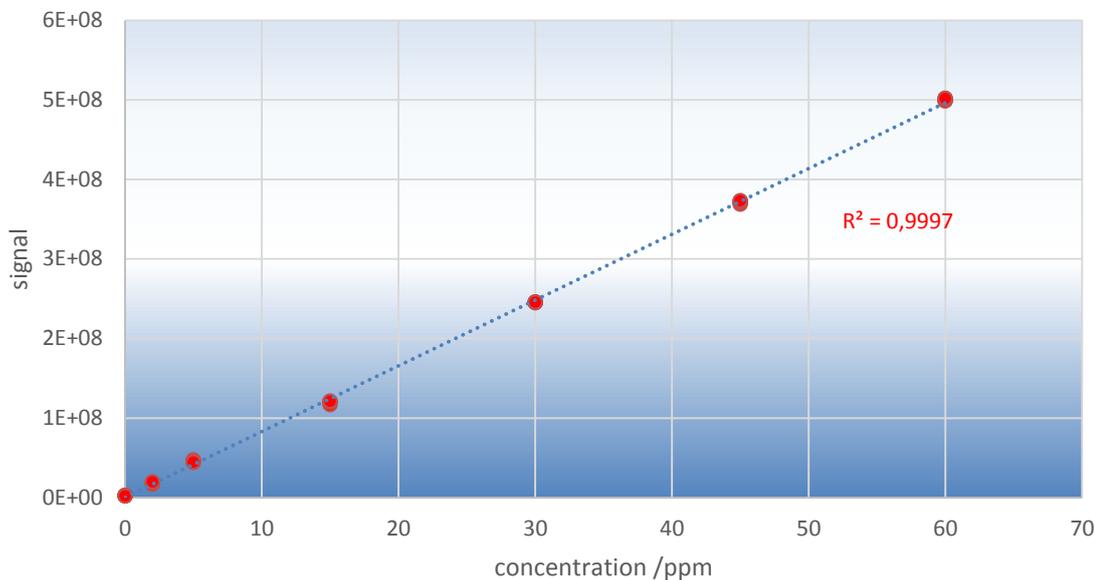
Hardware settings			
Ar/He	:	100 ml/min	Furnace (°C) : 1050
Pyro O2	:	450 ml/min	S PMT HV (V) : 600
Carrier O2	:	25 ml/min	
Gasmix carrier gas	:	30 ml/min	

**Table 2:** Analytical parameters of MultiTek



Obtained calibration curve shows perfect linearity.

**Figure 4:** Calibration curve



Concentration S, ppm	RSD (%)
60	0,3
45	0,5
30	0,1
15	1,7
5	2,8
2	8,0

### Quality control

A LPG with certified sulfur concentration at 5.7ppm is vaporized, injected and analyzed without dilution under the injection conditions of an unknown sample. The standard is regularly analyzed during two months without re-calibration or change of the test parameters. The differences between measurements are well below the repeatability requirements of the ASTM D6667 standard.



### Conclusion

The solution enables calibration of an elemental analyzer and checks the linearity of response without really limitation on the number of points. Equipped with an injection valve and flexible software, the system can interface with the analyzer and run fully automatic multipoint test sequences of routine quality assurance. Use a LPG standard avoids matrix effects (that may occur if an analyzer is calibrated with gases while samples being LPG), giving more accurate results. Using accurate diluter, buying multiple LPG standards is not necessary, having only one LPG standard at high concentration you prepare that you need on-site saving time and money. The solution offered by AlyTech is significantly increase productivity, safety and improve your quality assurance while running GPL samples.