APPLICATION NOTE LD24-04



Analysis of trace impurities in UHP Ammonia



Ammonia (NH3) is an important process chemical in semiconductor manufacturing. It is very commonly used for deposition of silicon nitride and is also used for nitridation or for deposition of other nitrides. Well known as chemical vapor deposition (CVD) and plasma etching processes.

Ammonia presents particular difficulties, since liquid ammonia contains both solid and volatile impurities, many of which are damaging to electronic components if present during the manufacturing process. The impurities level and content may vary widely depending on the source as well as the handling method, and all such impurities must be removed before the ammonia can be used in electronic component production lines.

To meet this standard, production facilities have had to obtain high-quality ammonia at considerable cost from the limited sources which are able to supply ammonia at an acceptable grade. Only qualified suppliers can be used, and new suppliers must be qualified before their product can be accepted.

A further constraint is presented by Department of Transportation regulations, under which aqueous ammonia must be shipped at ammonia concentrations no higher than 30%.

For these reasons, the ppb analysis of the impurities in NH3 is critical in the semiconductor facilities. The gas chromatography is a well known analysis technique to properly measure low ppb trace impurities in NH3 sample gas. The technique ensures no interference from the background gas and good sensitivity to the impurities to be measured.

LDETEK SOLUTION

The purity of ammonia can be qualified with the use of the MultiDetek3 gas chromatograph configured with PED.

The unit is configured with a measuring range of 0-5ppm and IdI of 3-5ppb for impurities H2-02-N2-C0-C02-CH4-C02-C2H6-C3H8-C4H10 in a sample gas UHP ammonia(NH3). The PED (plasma emission detector) has been mounted in the GC to measure the ppb impurities in UHP NH3 using Helium as carrier gas. All previously listed impurities are measured within one single analyser.

As the ammonia is listed as a very toxic gas, our GC configuration offers here a specific configuration of the sample flow path. A multiport selection valve mounted in a purged box which is purged with the helium carrier gas ensures the sample is not in contact with its surrounding air. The selection valve ensures to minimize the period which the ammonia is going into the injection's valves and the sample loops. A specific sequence is configured to have mostly helium in the GC sample flow path with a small period having the NH3 filling the sampling loops for proper analysis. The sequence is built in consequence to properly purged the loops prior to fill with ammonia sample gas. This solution offers the GC to have an improved safety level and have an extended lifetime due to the limited time of the analytical flow path in contact with the ammonia.

Other configurations and ranges/IdIs are possible. The parameters mostly depend on the site production requirements and process.

RESULTS

A series of chromatograms (Span calibration) of trace impurities H2-02-N2-C0-CH4-C02-C2H6-C3H8-C4H10 in balance gas UHP Ammonia (NH3)



Peak	Unit	Calibration Value _Area Counts	
		5.00	2205
HZ	ppm	5.00	2395
02	ppm	4.50	3459
N2	ppm	4.57	14408
со	ppm	4.96	12943
CH4	ppm	4.98	12387
CO2	ppm	4.88	6923



Peak	Unit	Calibration Value	_Area Counts	
C2H6	ppm	4.76	23976	
	ppm		20070	
C3H8	ppm	5.00	36574	
C4H10	ppm	4.76	38809	

Limit of detection (based on 3 times the noise level from a blank)

COMPONENTS	CONCENTRATION (ppm)	PEAK HEIGHT	NOISE	LDL (3X NOISE)
H2	5.0	2500mV	0.43mV	Зррb
02	4.5	2200mV	0.76mV	5ppb
N2	4.57	2291mV	0.51mV	3ppb
CO	4.96	2500mV	0.59mV	4ppb
CH4	4.98	2500mV	0.48mV	Зррb
CO2	4.88	2500mV	0.46mV	Зррb
C2H6	4.76	2400mV	0.71mV	4ppb
C3H8	5.00	2500mV	0.67mV	4ppb
C4H10	4.76	2460mV	0.69mV	4ppb

Note: other LDL could be obtained with different injection volume and chromatographic condition.

Repeatability: Based on the GC standards. Using 6 of 10 consecutive runs, being lower than 5% of 3*CV%

Linearity: Based on the GC standards. A linear curve having its R2 at a value between 0.998 and 1.00.

Accuracy: Based on the GC standards. <= 1% of error or IdI whichever is higher

The MultiDetek3 detailed in this application note complies with the previously listed repeatability/linearity and accuracy standards.

CONCLUSION

The MultiDetek3 configured with the PED module offers a good analytical solution for trace ppb/ppm impurities for the validation of the quality and the production for semiconductor Ammonia. The gas chromatograph is configured with standard industrial communication protocols and a remote-control interface. Due to its high sensitivity plasma emission detector, measuring trace impurities with the MultiDetek3 gas chromatograph down to sub ppb is perfectly suitable for the semiconductor ammonia. The MultiDetek3 is a very robust gas analyzer configured for industrial market to run 24/7. Combined with the other LDetek accessory modules, it fits the complete application requirements of the industry.



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