Application Note: 43031

Total Chlorine Analysis in Aromatic Hydrocarbons using the Thermo Scientific ECS Analyzer

Key Words

- Chloride
- Hydrocarbons
- ASTM

Introduction

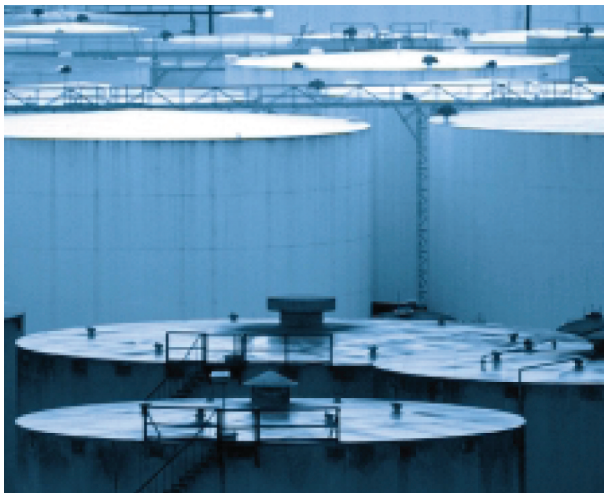
Aromatic hydrocarbons are light petrochemicals used as intermediate products within petrochemical cracking processes. The total chlorine content in for example benzene and p-xylene samples is tested to optimize the catalytic processes. Chlorine compounds will also cause corrosion of the piping and equipment within the chemical production process, with higher costs of operation as a result.

This application note describes the analysis performance of the Thermo Scientific ECS 3000 Total Chlorine Analyzer with the use of coulometric detection in aromatic hydrocarbon matrices.

Referenced Documents

The Thermo Scientific ECS 3000 Total Chlorine Analyzer complies with the following standard methods for this particular application:

ASTM D5808	Standard Test Method for Determination of Total Organic Chloride in Aromatic Hydrocarbons and Related Chemicals by Microcoulometric Detection
ASTM D5194	Standard Test Method for Trace Chloride in Liquid Aromatic Hydrocarbons



Principle of Operation

The benzene and p-xylene samples are automatically introduced into the oxygen rich and heated (1000 °C) combustion tube by use of the liquids sample introduction module and a syringe injection needle.

In this process, the bounded (organic) chlorides (OX) convert into inorganic chlorides (HCl). A sulfuric acid scrubber then conditions the combustion gases by removing water formed in the combustion process. The conditioned gases are directed into the titration cell which is filled with acetic acid electrolyte solution.

The electrodes are positioned in the cell and connected to the microcoulometer. Here, titration of the formed hydrochloric acid gas (HCl^-) with silver-ions (Ag^+) occurs.

System settings

Argon carrier gas	100 ml/min
Oxygen combustion gas	180 ml/min
Oxygen turbo gas	80 ml/min
Oxygen bypass gas	50 ml/min
Inlet temperature.	500 °C
Furnace temperature I	1000 °C
Furnace temperature II	1000 °C
Scrubber temperature	270 °C
Sample volume	100 µl

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Results of Chlorine Analysis

Standard	Q (µC)	Total Chloride (mg/kg)
4.5 ppm Cl	1262.7	4.64
4.5 ppm Cl	1259.7	4.63
4.5 ppm Cl	1268.3	4.66
4.5 ppm Cl	1250.9	4.60
4.5 ppm Cl	1266.1	4.65

Sample	Q (µC)	Total Chloride (mg/kg)
p-Xylene	273.2	1.00
p-Xylene	288.0	1.06
p-Xylene	278.1	1.02
p-Xylene	288.1	1.06
p-Xylene	277.6	1.02
p-Xylene	301.3	1.11

Sample	Q (µC)	Total Chloride (mg/kg)
Benzene I	77.8	0.29
Benzene I	70.9	0.26
Benzene I	71.1	0.26
Benzene I	61.6	0.23
Benzene I	66.2	0.24
Benzene I	71.4	0.26
Benzene II	23.0	0.08
Benzene II	24.7	0.09
Benzene II	24.6	0.09
Benzene II	25.8	0.09
Benzene II	26.1	0.10
Benzene II	25.1	0.09

Summary

Sample	Q (µC)	Average (mg/kg)	SD (mg/kg)	RSD (%)
4.5 ppm standard	1261.54	4.64	0.023	0.5
p-Xylene	284.38	1.05	0.040	3.8
Benzene I	69.52	0.26	0.021	8.0
Benzene II	24.88	0.09	0.006	7.0

N=6

Conclusion

The Thermo Scientific ECS 3000 is a high performance instrument for the analysis of low levels of chlorine in aromatic hydrocarbons. This is demonstrated by the excellent repeatability and accuracy obtained during this sample analysis.

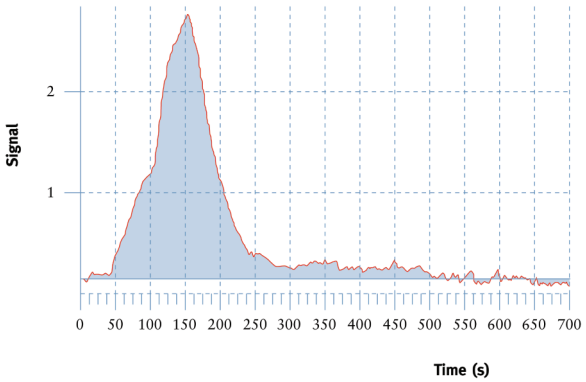

Figure 1: Example of analysis signal from the titration cell after combustion of a p-xylene sample.

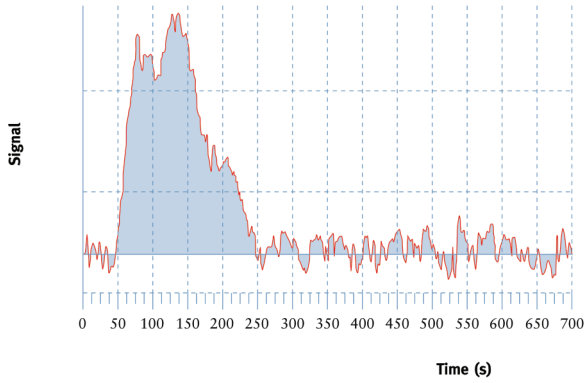

Figure 2: Example of analysis signal from the titration cell after the combustion of a benzene sample.

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