AQUASTAR®

Water Standard Oil:

A Novel Standard for Coulometric Karl Fischer Analysis of Oil Samples

Validation and control requirements for Karl Fischer (KF) instrumentation are specified in standard methods and regulatory guidelines, such as ASTM, GLP/GMP, ISO, etc. Typically, certified water standards traceable to a standard reference material (SRM) and specially formulated for this purpose are used. The use of such standards applies to direct KF titration methods, as well as to methods that involve using a KF Oven or an Oil Evaporator in conjunction with the titrator.

The vast majority of water standards currently used in KF are based on organic solvents, such as xylene or methoxybenzene. Therefore, the dissolution characteristics of these standards in KF solvents typically used in direct titration differ substantially from the dissolution characteristics of most petroleum product samples. Additionally, low-moisture KF standards for coulometry are currently available in the 50 ppm to 1000 ppm range, whereas the water content of many oil samples can be considerably lower.

Recently, through collaboration with the extensive research efforts of our parent company, Merck KGaA, (Darmstadt, Germany) and Mettler-Toledo, Inc. (Columbus, OH), EMD Chemicals has developed the **AQUASTAR® Water Standard Oil** (Cat. No. 1.88055.0010), an entirely novel NIST-traceable KF water standard, formulated specifically for the petrochemical industry. It is based on a transformer oil matrix, and has the lowest certified water content of any KF standard commercially available today.

This innovative new standard is directly traceable to SRM 2890 from NIST (National Institute of Standards and Technology). Each package of 10 sealed single-use 8 ml ampoules is shipped with the exact water content, accuracy, method traceability to the NIST batch, and minimum shelf life listed on the enclosed Certificate of Analysis.

Additionally, the new **AQUASTAR® Water Standard Oil** is specially formulated for both direct coulometric KF methods, where the oil sample is injected directly into the titration cell, and for indirect coulometric KF methods, which employ an Oil Evaporator in conjunction with a KF titrator.

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The table below illustrates the performance of the new **AQUASTAR® Water Standard Oil** in the Mettler DL39 coulometer equipped with a fritted cell as compared to a conventional xylene-based 100 ppm water standard. In the first trial, the standards were injected directly into the titration cell, while in the second trial, the standards were injected into the evaporation chamber of the **AQUASTAR® EV2000L Oil Evaporator** used in conjunction with the coulometer.

	AQUASTAR Water Standard Oil (1.88055.0010)	AQUASTAR Water Standard 0.01% (1.88050.0010)
CERTIFIED WATER CONTENT (PPM)	15±3	94±10
TRIAL 1 - DIRECT INJECTION INTO TITRATION CELL OF COULOMETER		
ANOLYTE CATHOLYTE	AQUASTAR Coulomat A (AX1697A) AQUASTAR Coulomat C (AX1697C)	AQUASTAR Coulomat A (AX1697A) AQUASTAR Coulomat C (AX1697C)
MEAN WATER CONTENT (PPM)	17.638	96.442
STANDARD DEVIATION (PPM)	0.527	0.976
REL. STANDARD DEVIATION (%)	2.99	1.01
TRIAL 2 - INJECTION INTO EVAPORATION CHAMBER OF OIL EVAPORATOR		
ANOLYTE CATHOLYTE	AQUASTAR Coulomat A (AX1697A) AQUASTAR Coulomat C (AX1697C)	AQUASTAR Coulomat A (AX1697A) AQUASTAR Coulomat C (AX1697C)
MEAN WATER CONTENT (PPM)	16.570	100.719
STANDARD DEVIATION (PPM)	1.343	2.774
REL. STANDARD DEVIATION (%)	8.10	2.75

INSTRUMENT: METTLER TOLEDO DL39 METHOD: I(pol) = 2 µA, U(stop) = 100 mV, Gen. rate = normal, Rel. drift stop: 3 µg/min

In conclusion, this innovative new standard features a number of distinct advantages over conventional standards:

- **Lowest available water content** for increased confidence in results in low ppm range
- **Transformer oil-based formulation** for increased confidence in results due to lack of sample vs. standard dissolution profile dissimilarities
- **NIST-traceability** to facilitate compliance with GMP/GLP, ISO, ASTM, etc. requirements
- **Universal suitability for titrators and Oil Evaporators** to allow the use of the same standard for both direct and evaporator-based KF methods

For additional **AQUASTAR®** product Technical and Applications Support call: **1-800-222-0342**