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Water determination in the process environment

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ProcessLab

Abstract

The atline system presented here enables water to be determined quickly and easily. It is of an appropriately robust design for the harsh production environment. Thanks to its modular structure it can be adapted to any particular process, and water contents between 0.02 and 50% can be determined in numerous intermediate and end products. All the analytical results are available for monitoring and control purposes and can be exported either via Ethernet or analog outputs on the I/O controller or processed further in a process control system.

Introduction

Many production processes require close monitoring of the water content. For many intermediate and end products the water content has to be maintained within narrowly defined limits. If sudden changes in water content are expected during a production process, it is necessary to resort to fast inline or online measuring techniques that are frequently both more complicated and more expensive. If different production lines or process areas need to be monitored, the use of an atline analysis system is recommended. Here the sample is taken manually and then fed into the system. In this way different samples from various process stages or units can easily be analyzed.

Karl Fischer water determination

Numerous methods exist for determining water. One of these is the extremely simple, but error-prone drying method using the drying oven or infrared lamp. Water content can also be determined titrimetrically or spectroscopically (e.g. Near InfraRed, NIR). The Karl Fischer titration (KFT) used in the atline analysis system is considered the most accurate and most reproducible water determination method. It determines not only free water, but also the water that is adsorbed on the surface or trapped in the crystal structure. In developing his new analytical method, Karl Fischer (1) started out from the well known Bunsen reaction for determining sulfur dioxide in an aqueous solution:

$$SO_2 + I_2 + 2\,H_2O \rightleftharpoons H_2SO_4 + 2\,HI$$

Using an excess of SO_2 in the presence of bases this reaction can also be used to determine water. In this way the classic KF reagent, a solution of iodine and sulfur dioxide in a buffered mixture of pyridine and methanol (2), came into being. Since being discovered more than seventy years ago, KFT has experienced unparalleled success. As a result of constant improvements in the commercially available KF reagents, there are now various one- and two-component reagents available for different matrices and water contents.

Apart from volumetric Karl Fischer titration, there is also the possibility of generating the iodine required for reaction coulometrically with the help of electrical current directly in the measuring cell. Whereas coulometric water determination is suitable for determining very small water contents, the volumetric KFT applied in this work is used to determine higher water contents (2-3).

KFT can be used for the determination of water in oils and biofuels, salts, surfactants, food, cosmetics and in pharmaceutical raw materials and final products

The analysis system at a glance

Like all ProcessLab atline analysis systems, the one presented here is meant for direct use in the production area. The system is of an appropriately robust design for production and automatically determines water content by means of KFT.

Each analysis system consists of an analysis module and an operating unit with an optional Touch monitor (Fig. 1).

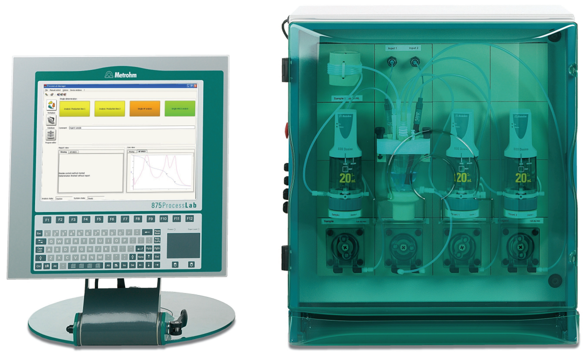

Fig. 1: Analysis module and operating unit with optional Touch monitor

Both the analysis module and the operating unit are contained in a robust, splashproof housing and are therefore ideally equipped for use in harsh production environments. ProcessLab is an analysis system with a modular structure throughout that can be adapted easily to any particular process conditions.

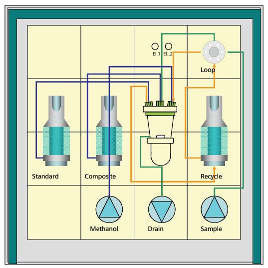

Fig. 2: Analysis module

The system consists of a clearly structured analysis module (Fig. 2). It contains a measuring vessel with magnetic stirrer, a sample loop for automatic metering of the sample, one Dosino for exact dosing of the reagent (Hydranal® Composite) and of the water standard for titer determination plus one Dosino for the conditioned solvent. Peristaltic pumps enable automatic addition of methanol as well as automatic cleaning of the measuring vessel. Thus it is possible to determine the water content easily and fully automatically within a few minutes.

All analytical results are saved in a shared database and are available for monitoring and control purposes. The data can be exported either via Ethernet or analog outputs on the I/O controller or supplied to a process control system.

Water determination at the process line

The atline analysis system presented here was used to determine the water content in solvents such as acetonitrile, phenol, methanol and isopropanol as well as in window cleaner solutions. Ten repeat measurements are shown for each of the different substances together with the overall reproducibility achieved with the system (Tab. 1).

Tab. 1: Determination of water content in different substances

Matrix	Acetonitrile	Phenol/methanol	Methanol	Isopropanol	Window cleaner
Water content (w/w)	≤ 0.2%	-*	2.5%	10%	-*
KFT	[%]	[%]	[%]	[%]	[%]
1	0.0411	0.670	2.540	10.04	36.27
2	0.0413	0.673	2.558	10.05	36.26
3	0.0412	0.675	2.540	10.04	36.26
4	0.0413	0.669	2.559	10.05	36.29
5	0.0410	0.674	2.541	10.02	36.23
6	0.0412	0.672	2.544	9.99	36.17
7	0.0409	0.669	2.543	10.00	36.15
8	0.0412	0.675	2.530	10.03	36.15
9	0.0410	0.678	2.529	10.04	36.27
10	0.0415	0.676	2.544	9.98	36.30
Mean [%]	0.0412	0.673	2.543	10.02	36.24
RSD [%]	0.44	0.46	0.39	0.25	0.16

*water content not specified before KFT

Variation of sample volumes, reagent concentration and dosing units allows to adjust to the determination range of the particular application and thus to determine very low as well as very high water contents (Tab. 2).

Tab. 2: Volumes of sample loop and dosing unit as well as concentrations of the KF reagent used for different water contents

Water content	Volume		Concentration of the
	Sample loop	Dosing unit	Hydranal®-Composite reagent
[%]	[mL]		[mg/mL]
0.02...0.20	5	5	2
0.05...0.50	5	5	5
0.20...2.00	5	20	5
0.50...5.00	1	10	5
1.00...10.0	0.5	10	5
2.50...25.0	0.2	10	5
5.00...50.0	0.2	20	5

References

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