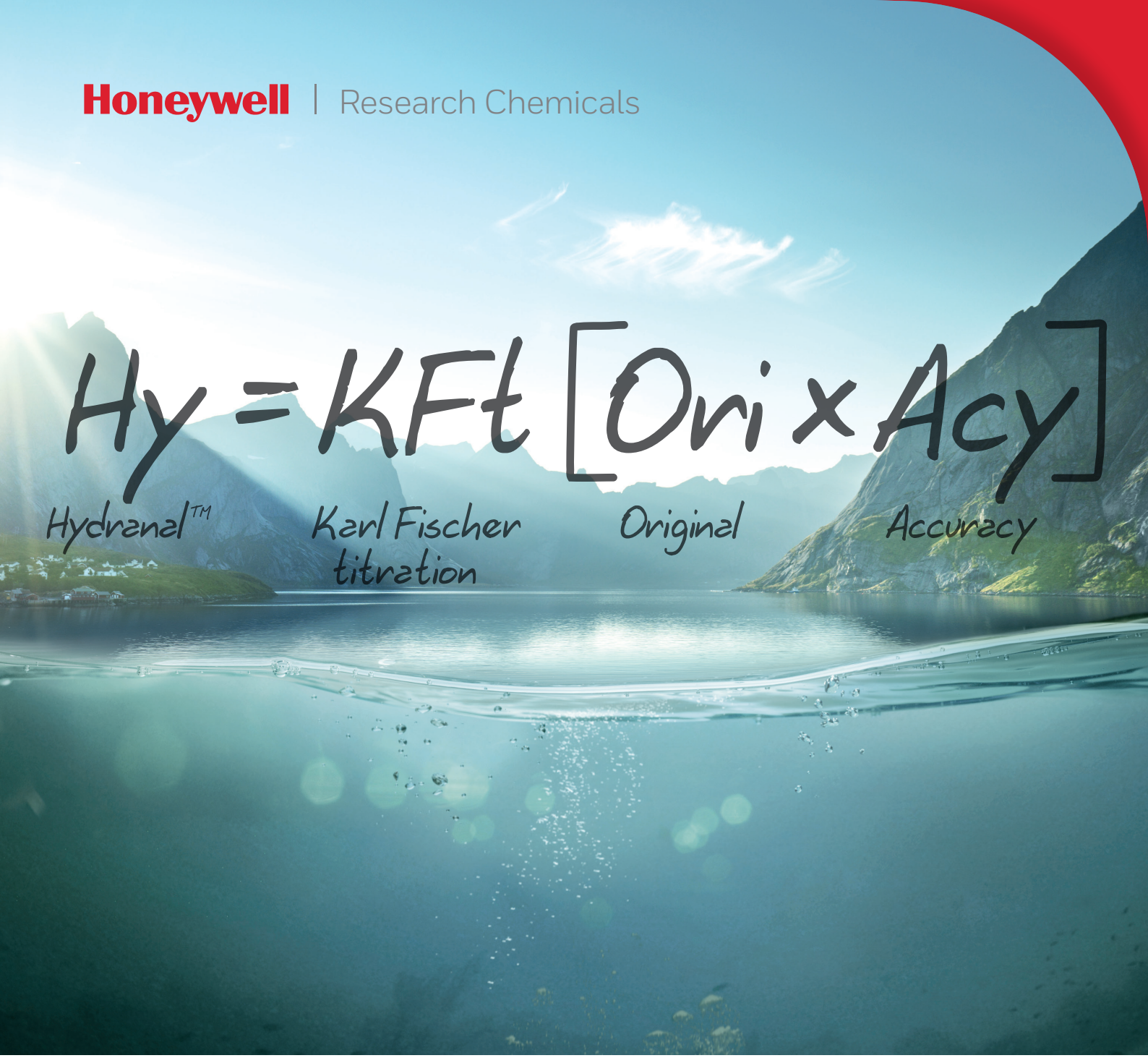
Honeywell | Research Chemicals



Discover the perfect formula

HYDRANAL™ Product Overview Guide

Reagents for water determination by Karl Fischer titration

Greyhound CHROMATOGRAPHY AND ALLIED CHEMICALS

HYDRANAL™ Reagents from Honeywell Research Chemicals

Water content can affect product quality, texture, shelf life, chemical stability and reactivity. Karl Fischer titration is a universally accepted method for measuring water content in all types of substances, including chemicals, oils, pharmaceuticals and food. In 1979, researcher Dr. Eugen Scholz improved Karl Fischer titration by replacing noxious pyridine with imidazole. This innovation became the foundation of Hydranal™, the world's leading pyridine-free reagents for Karl Fischer titration.

From Dr. E. Scholz's pioneering research to the ongoing product improvements of today, Honeywell offers a wide range of Karl Fischer reagents for both volumetric and coulometric titrations for nearly all types of samples, completed by a broad range of standards.

With the inclusion of Fluka™ into Honeywell Research Chemicals, Hydranal became an important part of the overall product portfolio. Hydranal reagents and water standards have always been developed and produced in our plant in Seelze, Germany, meaning you can be sure to enjoy the same composition, quality, service and technical support you always have.

Honeywell is your partner for reliable and easy-to-use pyridine-free Karl Fischer reagents

Advantages of HYDRANAL Reagents:

- High titration speed
- Stable end points
- Accurate results
- Long shelf life
- Wide applicability
- World leading technical support

HYDRANAL Product Line at a Glance

PRODUCT LINE	PRODUCT DESCRIPTION
HYDRANAL-Composite	The most flexible and commonly used reagents for one-component volumetric titration
HYDRANAL Special Media	Special reagents like Methanol Rapid, E-Types and K-Types
HYDRANAL-Titrant/Solvent	Reagents for two-component volumetric titration
HYDRANAL-Coulomat	Reagents for coulometric titration for samples with low water content
HYDRANAL-Water Standards	Standards with verified water content for titer determination, monitoring precision, accuracy, validation and inspection of Karl Fischer titrators
HYDRANAL-CRM Water Standards	Certified Reference Materials for titer determination, monitoring precision, accuracy, validation and inspection of Karl Fischer titrators

The Chemistry of Karl Fischer Titration

The Karl Fischer technique for water determination, invented in 1935 by Karl Fischer, is a titration based on the Bunsen reaction. In 1979 it was postulated by Dr. E. Scholz as a two-step equation:

(1) $ROH + SO_2 + R'N \leftrightarrow [R'NH]SO_3R$

(2) $[R'NH]SO_3R + H_2O + I_2 + 2R'N \rightarrow 2[R'NH]I + [R'NH]SO_4R$

ROH = alcohol, typically methanol
R'N = base

The oxidation of alkylsulfite to alkylsulfate in reaction (2) consumes water, which ideally comes only from the sample. Since water and iodine are consumed in a 1:1 stoichiometric ratio, the amount of water in the original sample is calculated by the amount of iodine required to complete the reaction. The iodine is measured either volumetrically directly by volume or coulometrically by the amount of current required for the generation of iodine.

How the Base Affects Reaction Kinetics

The type of base (R'N) and its concentration affect the overall reaction rate. Traditionally, pyridine was used as the base. However, because of its weak basicity, pyridine cannot completely neutralize the alkyl-sulfurous acid intermediate. As a result, reaction (1) is slow, does not go to completion and the end point is not stable. Because of this lack of stability, the repeatability of the results is often very poor. In addition, pyridine has a noxious odor.

Imidazole and 2-Methylimidazole as Alternatives to Pyridine

Dr. E. Scholz and his research team sought to replace the pyridine with a stronger base with a higher affinity for the alkylsulfite. Imidazole was found to have even more benefits than pyridine besides not having the noxious odor. Imidazole allows reaction (1) to go to completion rapidly and provides a stable end point. Later on researchers found that adding a second base, 2-methylimidazole, to the imidazole, enhances stability and reduces the appearance of undesired crystallization.

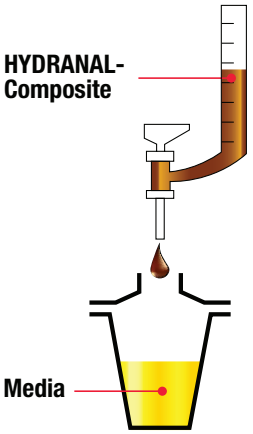
Volumetric One-Component Titration – Titrating Agents

HYDRANAL-Composite

Hydranal-Composite is the world's most frequently used pyridine-free Karl Fischer reagent. This one-component reagent has been proving its capabilities in volumetric titration for more than thirty five years in a large range of applications in the most diverse fields of research and industry. Ongoing development work has achieved significant improvements to this reagent.

Advantages of HYDRANAL One-Component Reagents:

- Unlimited water capacity
- Convenient and easy to use
- The greatest flexibility in working media selection
- Suitable for methanol-reacting compounds, e.g. ketones and aldehydes
- Long shelf life (three years)



Improved Composition

Hydranal-Composite contains all the reactants including iodine, sulfur dioxide, and the bases imidazole and 2-methylimidazole, dissolved in diethylene glycol monoethyl ether (DEGEE). Adding 2-methylimidazole in addition to imidazole improves the stability and eliminates the formation of crystals which can interfere with the titrator's performance. The crystallization of the reagent was occasionally observed under the influence of airborne moisture and also after prolonged residence of the reagent in the Karl Fischer titrator's tube system. This effect is prevented by a new and improved formulation.

Enhancement of Titer Stability

When comparing the old and new formulation it becomes obvious that the new formulation is significantly more stable with a loss of concentration less than 5% per year vs. approx. 10% for the old formulation. Hydranal-Composite is additionally stabilized with DEGEE as a solvent. The results of the tests into titer decline are shown in Figure 1.

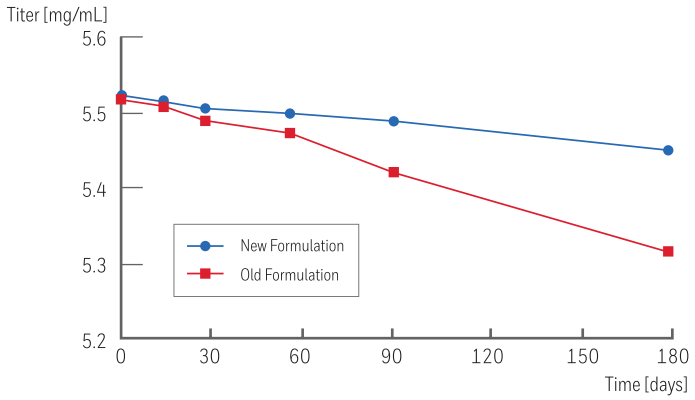


Figure 1. Results of the titer stability tests

PRODUCT NUMBER	PRODUCT NAME	DESCRIPTION	PACKAGING
34827	HYDRANAL-Composite 1	One-component reagent, titer ~1 mg/mL	500 mL; 1 L
34806	HYDRANAL-Composite 2	One-component reagent, titer ~2 mg/mL	500 mL; 1 L; 2.5 L
34805	HYDRANAL-Composite 5	One-component reagent, titer ~5 mg/mL	500 mL; 1 L; 2.5 L
34816	HYDRANAL-Composite 5 K	One-component reagent for titration of ketones and aldehydes, titer ~5 mg/mL	500 mL; 1 L; 2.5 L

Volumetric One-Component Titration – Media

With one-component reagents the medium (i.e. the solvent required) is chosen according to the dissolution and chemical properties of the sample substance being analyzed. The most commonly used medium is dry methanol.

The speed, time taken and accuracy of the Karl Fischer reaction is influenced by the medium used in the titration vessel. The Hydranal-Composite, one-component reagents, are already buffered to an optimum of pH by using imidazoles. Thus the performance of the titrating agent is optimized to ensure a rapid Karl Fischer titration, however, there is still room for improvement on the use of the solvent.

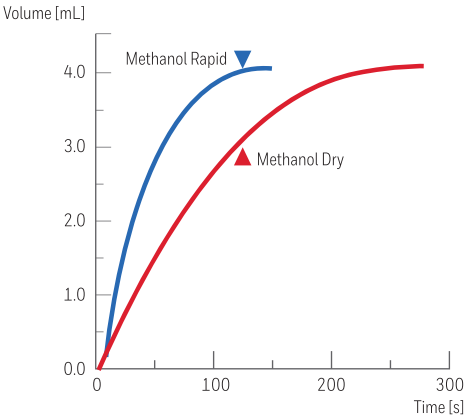


Figure 2. Titration of 20 mg water

HYDRANAL-Methanol Rapid

Methanol is the most commonly used medium in the titration vessel, however it is an unbuffered solvent. When using Hydranal-Methanol Rapid, you will see a clear improvement in speed and accuracy of the titration. This is due to the accelerators in the medium, which are unique to Hydranal-Methanol Rapid and enable an optimal Karl Fischer titration (see Figure 2).

Advantages of HYDRANAL-Methanol Rapid:

- Much shorter titration time
- Rapid end point
- High accuracy of the analysis

HYDRANAL-CompoSolver E

In case a less toxic solvent is preferred, Hydranal-CompoSolver E, an ethanol based medium, has a similar performance to Hydranal-Methanol Rapid.

HYDRANAL-Solver (premixed)

Many non-polar samples (e.g. oils, fats, organic components) appear with a poor solubility in methanol and require the addition of a solubilizer. To overcome these challenges, a series of specially designed media has been developed based on the most suitable solvent mix.

HYDRANAL-K Media

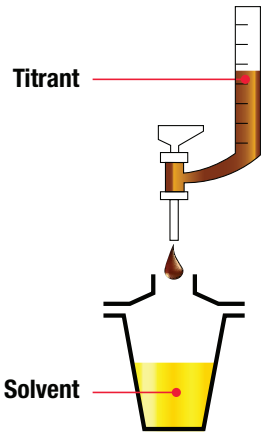
For compounds reacting with methanol, like ketones and aldehydes, three different media have been developed. Comparing the three media based on their toxicity and capacity to suppress side effects, we recommend the use of Hydranal-Medium K as the first choice.

PRODUCT NUMBER	PRODUCT NAME	DESCRIPTION	PACKAGING
37817	HYDRANAL-Methanol Rapid	Medium containing accelerators	1 L; 2.5 L
34741	HYDRANAL-Methanol Dry	Medium for general use	1 L; 2.5 L
34734	HYDRANAL-CompoSolver E	Ethanol-based medium containing accelerators	1 L; 2.5 L
34697	HYDRANAL-Solver (Crude) Oil	Working medium containing methanol, xylene and chloroform for titration in oils	1 L; 2.5 L
37855	HYDRANAL-LipoSolver CM	Working medium containing methanol and chloroform for titration in non-polar samples	1 L
37856	HYDRANAL-LipoSolver MH	Working medium containing methanol and 1-hexanol for titration in non-polar samples	1 L
34698	HYDRANAL-Medium K	Less toxic working medium containing chloroform for ketones and aldehydes	1 L
34738	HYDRANAL-KetoSolver	Working medium free of halogenated solvents for ketones and aldehydes	500 mL; 1 L
34817	HYDRANAL-Working Medium K	Working medium containing chloroform and 2-chloroethanol for ketones and aldehydes	1 L

Volumetric Two-Component Titration

HYDRANAL-Titrant/Solvent

Two-component reagent have been developed to use the stability advantage of a pure alcoholic solution of iodine. Additionally the buffered system provides a very fast titration performance.



Composition

In two-component reagents the Karl Fisher reactants are separated into two solutions: the titrant and the solvent. Hydranal-Titrant contains iodine dissolved in an alcohol with a precisely defined concentration. Hydranal-Solvent is an alcoholic solution of sulfur dioxide and imidazole.

The alcohol is either methanol for standard reagents or ethanol for E-type reagents.

Further types of Solvent reagent based on different mixtures enable to meet the sample dissolution properties.

Advantages of HYDRANAL Two-Component Reagents:

- High titration speed
- Ideal accuracy for small amounts of water
- High buffer capacity
- Exact and stable titer
- E-type reagents: reduced toxicity compared to methanol
- Long shelf life (three years for titrants, five years for solvents)

PRODUCT NUMBER	PRODUCT NAME	DESCRIPTION	PACKAGING
		METHANOL BASED	
34811	HYDRANAL-Titrant 2	Two-component reagent, titer ~2 mg/mL	500 mL; 1 L; 2.5 L
34801	HYDRANAL-Titrant 5	Two-component reagent, titer ~5 mg/mL	500 mL; 1 L; 2.5 L
34800	HYDRANAL-Solvent	Working medium for two-component titration	1 L; 2.5 L
		ETHANOL BASED	
34723	HYDRANAL-Titrant 2 E	Two-component reagent, titer ~2 mg/mL	1 L
34732	HYDRANAL-Titrant 5 E	Two-component reagent, titer ~5 mg/mL	500 mL; 1 L; 2.5 L
34730	HYDRANAL-Solvent E	Working medium for two-component titration	500 mL; 1 L; 2.5 L
		SPECIAL MEDIA	
34812	HYDRANAL-Solvent CM	Working medium for two-component titration, containing methanol and chloroform for titration in non-polar samples	1 L; 2.5 L
34749	HYDRANAL-Solvent Oil	Working medium for two-component titration, containing methanol and 1-hexanol for titration in non-polar samples	1 L
34697	HYDRANAL-Solver (Crude) Oil	Working medium containing methanol, xylene and chloroform for titration in oils	1 L; 2.5 L

Coulometric Titration

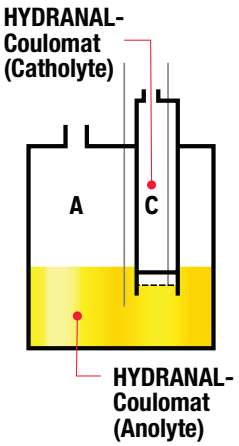
HYDRANAL-Coulomat

Coulometric Karl Fischer titrations normally require two reagent solutions: an anolyte (the solution in the anodic compartment) and a catholyte (the solution in the cathodic compartment). Hydranal-Coulomat A-type or E-type reagents are used as anolytes. The anolytes contain iodide and a sulfur dioxide/imidazole buffer in suitable solvents. Hydranal-Coulomat CG reagents are used as catholytes.

Coulometric reagents based on different solvent compositions serve to support the broad variety of samples analyzed, i.e. Hydranal-Coulomat Oil contains methanol, xylene and chloroform for titration in oils or methanol-free Hydranal-Coulomat AK for titration of ketones to suppress negative side effects.Furthermore, some working techniques are supported by special reagents i.e. the use of Karl Fischer oven by Hydranal-Coulomat AG-Oven or the use of a cell without diaphragm by Hydranal-Coulomat AD.

Coulometric Cells

There are two different types of coulometric cells: those with and those without a diaphragm. The diaphragm separates the anode chamber from the cathode chamber. Oxidation of I^- to I^2 occurs at the anode, whereas the reduction of protons to H^2 occurs at the cathode. For cells without a diaphragm the anodic and cathodic compartments are not separated and only one reagent, the anolyte, is needed. Though the latter coulometric cell may seem more convenient to use, the cell with diaphragm achieves the highest accuracy down to the trace range of water. Recommendation details are given in description below the table.



Advantages of HYDRANAL Coulometric Reagents:

- Easy to use
- High accuracy for trace amounts of water
- Stable conditions of the titration vessel
- Broad product range
- Long shelf life (up to five years)

PRODUCT NUMBER	PRODUCT NAME	DESCRIPTION	PACKAGING
34807	HYDRANAL-Coulomat A	Anolyte preferred for cells with diaphragm*	500 mL
34836	HYDRANAL-Coulomat AG	Anolyte suitable for cells with and without diaphragm	500 mL; 1 L
34843	HYDRANAL-Coulomat AG-H	Anolyte for titration of long-chained hydrocarbons, preferred for cells with diaphragm*	500 mL
34739	HYDRANAL-Coulomat AG-Oven	Anolyte for determination with Karl Fischer oven, suitable for cells with and without diaphragm	500 mL
34820	HYDRANAL-Coulomat AK	Anolyte for titration of ketones, preferred for cells with diaphragm*	500 mL
34868	HYDRANAL-Coulomat Oil	Anolyte for titration of oils, preferred for cells with diaphragm*	100 mL; 500 mL
34726	HYDRANAL-Coulomat E	Anolyte based on ethanol, suitable for cells with and without diaphragm	500 mL
34810	HYDRANAL-Coulomat AD	Anolyte preferred for cells without diaphragm	500 mL
34840	HYDRANAL-Coulomat CG	Catholyte	10 x 5 mL
34821	HYDRANAL-Coulomat CG-K	Catholyte for titration of ketones	10 x 5 mL

* In theory all Hydranal-Coulomat anolytes may be used with either type of generator electrode: with or without a diaphragm. However, the anolytes which contain a co-solvent in addition to methanol show increased recoveries when used with a diaphragmless generator electrode. Therefore we recommend using a diaphragm generator electrode when working with a co-solvent containing anolyte. This will require the use of the appropriate catholyte.

Titer Standardization and Instrument Inspection

HYDRANAL-Water Standards

Quality management plays an important role in Karl Fischer titration. Calibration, validation and inspection of analytical instruments and reagents is performed with a specific amount of water, either pure water or water standards. The challenge with pure water is the low amount required (10-50 mg for volumetry, and 0.1-1 mg for coulometry), which is difficult to handle and weigh.

We therefore recommend Hydranal-Water Standards with an exactly confirmed water content for:

- Titer determination
- Monitoring precision and accuracy
- Validation and inspection of Karl Fischer titrators according to ISO, GMP, GLP and FDA guidelines

Traceability to a national standard or to a SI unit is often required in these guidelines. All Hydranal-Water Standards are tested against external reference material from national metrological institutions as the NIST (National Institute of Standards and Technology, USA) standard reference material SRM 2890, Water Saturated Octanol or CRM 4222 Water in Mesitylene from National Metrology Institute of Japan (NMIJ). Additionally they are calibrated based on high purity water.

Liquid standards consist of a solvent mixture with specific composition and precisely determined water content. They are packaged in glass ampoules under argon. Each box contains ten single-use ampoules which are easy to open (pre-notched).

Solid standards contain defined amounts of chemically bound water suitable for both general use as well as for the Karl Fischer oven. These standards are packed in amber glass bottles.

Advantages of HYDRANAL-Water Standards:

- Broad product range for volumetric and coulometric Karl Fischer applications
- Manufactured according to current ISO requirements
- Tested against NIST SRM 2890
- Long shelf life (up to five years)
- Convenient packaging
- Supplied with detailed instruction for use
- Report of Analysis showing the exact water content is included

HYDRANAL-CRM Water Standards

In 2014, Hydranal Technical Service in Seelze completed its combined accreditation according to ISO/IEC 17025 and ISO Guide 34, the so-called “Gold Standard Accreditation”, which is the highest achievable quality level for producers of Certified Reference Materials (CRMs). With the double accreditation, Hydranal introduced the very first commercially available CRM Water Standards for Karl Fischer titration.

PRODUCT NUMBER	PRODUCT NAME	DESCRIPTION	PACKAGING
34425	HYDRANAL-CRM Water Standard 10.0	Liquid CRM standard, water content 10.0 mg/g = 1.0%	10 x 8 mL
34426	HYDRANAL-CRM Water Standard 1.0	Liquid CRM standard, water content 1.0 mg/g = 0.1%	10 x 4 mL
34424	HYDRANAL-CRM Sodium Tartrate Dihydrate	Solid CRM standard, water content ~15.66%	10 g
34849	HYDRANAL-Water Standard 10.0	Liquid standard, water content 10.0 mg/g = 1.0%	10 x 8 mL
34828	HYDRANAL-Water Standard 1.0	Liquid standard, water content 1.0 mg/g = 0.1%	10 x 4 mL
34847	HYDRANAL-Water Standard 0.1	Liquid standard, water content 0.1 mg/g = 0.01% (shelf life 2 years, to be stored at 2-8°C)	10 x 4 mL
34446	HYDRANAL-Water Standard 0.1 PC	Liquid standard water content 0.1 mg/g = 0.01% (improved stability compared to 34847: shelf life 5 years, to be stored at room temp.)	10 x 4 mL
34694	HYDRANAL-Water Standard Oil	Liquid standard based on mineral oil, water content approx. 10 ppm (0.001%)	10 x 8 mL
34696	HYDRANAL-Standard Sodium Tartrate Dihydrate	Solid standard, water content ~15.66%	25 g
34693	HYDRANAL-Water Standard KF Oven 140-160°C	Solid standard for control of Karl Fischer ovens, water content ~5%, based on lactose	10 g
34748	HYDRANAL-Water Standard KF Oven 220-230°C	Solid standard for control of Karl Fischer ovens, water content ~5.55%, based on potassium citrate	10 g

Auxiliaries for Karl Fischer Titration

Karl Fischer titration is applied to multifarious substances. The nuances in sample properties influence the Karl Fischer titration differently. There are a number of ways to adjust the working conditions.

Solubilizers

In special cases the addition of solubilizer is required in order to enable a direct titration of the sample and avoid complicated and error-prone pre-dissolution and pre-extraction steps.

Buffers

The Karl Fischer reaction is pH dependant, with pH 5-7.5 being the ideal range. Strongly acidic samples slow the reaction and must be neutralized without inducing an alkaline reaction of the working medium prior to starting the titration. Strong bases can increase the pH of the working solution if the basicity exceeds the buffering capacity of the reagent. A titration end point will not be reached. Strong bases also must be neutralized prior to starting the titration.

Drying agents

Special drying agents are suitable to hold the near environment of the Karl Fischer equipment on a low water level or to dry carrier gases in case of oven technique.

HYDRANAL-Moisture Test Kit

For rough measurements without a titrator, special test kits for visual water determination according to Karl Fischer can be used. The set contains syringes, titration vessel and reagents: 2 x 500 mL Hydranal-Solvent E (34730), 100 mL Hydranal-Titrant 5 E (34732) and 100 mL Hydranal-Standard 5.0 (34813). Refills can be ordered separately.

PRODUCT NUMBER	PRODUCT NAME	DESCRIPTION	PACKAGING
34724	HYDRANAL-Formamide Dry	Solubilizer, max. 0.02% water	1 L
37863	HYDRANAL-Chloroform	Solubilizer, max. 0.01% water	1 L
37866	HYDRANAL-Xylene	Solubilizer, max. 0.02% water	1 L
34804	HYDRANAL-Buffer for Acids	Liquid buffer medium, based on imidazole	500 mL
37859	HYDRANAL-Buffer for Bases	Liquid buffer medium, based on salicylic acid	1 L
32035	HYDRANAL-Benzoic Acid	Buffer substance	500 g
37865	HYDRANAL-Salicylic Acid	Buffer substance	500 g
37864	HYDRANAL-Imidazole	Buffer substance	500 g
34813	HYDRANAL-Standard 5.0	Test solution for volumetric titration, water content 5.00 mg/mL	100 mL; 500 mL
34803	HYDRANAL-Sodium Tartrate Dihydrate	Test substance for volumetric titration, water content ~15.66%	100 g
34802	HYDRANAL-Water-in-Methanol 5.0	Reagent for volumetric back titration, water content 5.00 mg/mL	500 mL; 1 L
34788	HYDRANAL-Humidity Absorber	Drying agent for air and gases with indicator	500 g; 1 kg
34241	HYDRANAL-Molecular Sieve 0.3 nm	Drying agent for air and gases	250 g
37858	HYDRANAL-Moisture Test Kit	Test kit for the visual water determination according to Karl Fischer without titrator	1 kit

Technical Support

For more than 35 years, the Hydranal Technical Service Team has been gathering extensive and unmet experience and insights into Karl Fischer titration and its related challenges.

If you are looking to improve your Karl Fischer titration performance, the team of Hydranal experts can support you with:

- Selecting the most suitable Karl Fischer reagents for your samples
- Recommending application methods
- Troubleshooting technical problems (solubility, side reactions, etc.)
- Technical Karl Fischer seminars and trainings
- Comprehensive literature

To learn more about Hydranal reagents, visit hydranal-honeywell.com

Resources:	lab-honeywell.com/resources
Laboratory Reports:	lab-honeywell.com/hydranal-search
Events:	lab-honeywell.com/events
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Water determination in dimethyl sulfoxide (DMSO)

HYDRANAL™ Laboratory Report L 141

It is not possible to reproduce water determination in DMSO because it alters the stoichiometry of the Karl Fischer (KF) reaction. This shortcoming becomes even greater as the number of samples increases.

We tested the water content in different DMSO sample quantities according to the following procedure. 30 mL of Hydranal-Methanol Rapid, Hydranal-Methanol dry or Hydranal-CompoSolver E were introduced into the titration vessel and titrated to dryness with Hydranal-Composite 5. The sample (or sample plus water) was then added and titrated with Hydranal-Composite 5.

Results are compared in the tables below.

Table 1. Water determination in various DMSO sample quantities

DMSO SAMPLE VOLUME	HYDRANAL-COMPOSITE 5 CONSUMPTION	WATER QUANTITY DETECTED	WATER CONTENT DETECTED
1.0 mL	0.08 mL	0.44 mg	0.044%
2.0 mL	0.14 mL	0.77 mg	0.038%
5.0 mL	0.19 mL	1.05 mg	0.021%
10.0 mL	0.35 mL	1.93 mg	0.019%
20.0 mL	0.62 mL	3.41 mg	0.017%

Table 2. Different sample quantities of DMSO added to a defined water quantity (20.0 mg)

SAMPLE	HYDRANAL-COMPOSITE 5 CONSUMPTION	WATER QUANTITY DETECTED	RECOVERY RATE
20 mg H_2O	3.81 mL	20.00 mg	100.0%
20 mg H_2O + 1.0 mL DMSO	3.80 mL	19.95 mg	99.75%
20 mg H_2O + 2.0 mL DMSO	3.77 mL	19.79 mg	98.95%
20 mg H_2O + 5.0 mL DMSO	3.71 mL	19.48 mg	97.40%
20 mg H_2O + 10.0 mL DMSO	3.49 mL	18.32 mg	91.60%
20 mg H_2O + 20.0 mL DMSO	3.38 mL	17.74 mg	88.70%

Coulometric tests gave comparable findings:

- Water content detected in 1 mL DMSO: 311 ppm
- Water content detected in 20 mL DMSO: 266 ppm

Control conditions with Hydranal-Water Standard 1.0 (added water quantity: 1000 ppm) in the presence of DMSO produced the following results:

- Water content in the presence of 1 mL DMSO: 994 ppm
- Water content in the presence of 20 mL DMSO: 910 ppm

The volumetric and coulometric water content determination tests were carried out with two different samples.

The problem was picked up again in a series of subsequent tests. We ascertained that it is also impossible to perform indirect determination in a KF oven. A temperature ramp from 50°C to 250°C showed that the water is released by azeotropic distillation at temperatures between 130°C and 190°C. It is impossible to separate the water from the DMSO. The titration vessel is influenced in the same way as by direct injection of the sample. The error is comparable to the effect described for direct injection.

Conclusion

As a result of the various facts, the recommendation must be made that no more than 1 mL of DMSO should be analyzed. The injection of several samples in a coulometric cell should be avoided. Addition of a 1000 ppm standard shows the current effect on the titration vessel.

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VOLUMETRIC REAGENTS

34805	HYDRANAL-Composite 5	34734	HYDRANAL-CompoSolver E
34741	HYDRANAL-Methanol dry	37817	HYDRANAL-Methanol Rapid

WATER STANDARDS

34828	HYDRANAL-Water Standard 1.0	34426	HYDRANAL-CRM Water Standard 1.0

AUXILIARIES

34241	HYDRANAL-Molecular Sieve 0.3 nm	34788	HYDRANAL-Humidity Absorber

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Water determination in aldehydes and ketones

HYDRANAL™ Laboratory Report L 676

(Extract from HYDRANAL Manual, chapter 9.6)

Both aldehydes and ketones pose problems with Karl Fischer titration because they form acetals and ketals respectively with conventional KF reagents (Figure 9.6.a). The reaction forms water, which is also titrated, resulting in vanishing end points and erroneously high water content. With aldehydes a second side reaction, the bisulfite addition, can also occur (Figure 9.6.b). This reaction consumes water and leads to an erroneously low water content.

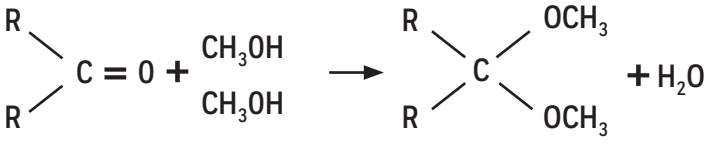

Figure 9.6.a. The formation of acetals or ketals.

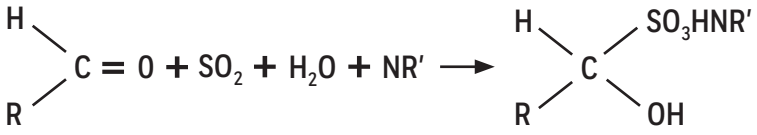

Figure 9.6.b. The bisulfite addition. NR' = base

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We have investigated the behavior of certain aldehydes and ketones toward the KF titration. The reactivity of aliphatic ketones decreases with increasing chain length. Aromatic ketones are less reactive than aliphatic ketones. Aldehydes are much more reactive than ketones and their tendency to undergo the bisulfite addition is particularly strong.

The formation of acetals and ketals can be suppressed by replacing methanol in the titrating agent with another solvent, typically pyridine or 2-methoxyethanol (methylglycol). However, we found both of these solvents to be unsatisfactory. Pure pyridine alters the stoichiometry of the KF reaction, enhances the bisulfite addition, and leads to a falsely low water content. 2 methoxyethanol does not sufficiently inhibit the formation of both ketals and acetals and results in a slow titration rate. The levels of water are too high and, because only small samples can be analyzed, the accuracy of the titration is negatively affected.

Our research identified suitable solvents that permit determination of water in aldehydes and ketones without adverse side reactions. These solvents are the basis of the Hydranal™ K-type reagents.

9.6.1 Volumetric titration

As a result of the challenges with KF titration of aldehydes and ketones, we developed special reagents for their determination by volumetric titration:

- Hydranal-Composite 5 K
- Hydranal-Working Medium K
- Hydranal-Medium K
- Hydranal-KetoSolver

The following abbreviated working procedure provides an introduction to their usage.

Procedure 9.6.1.1 Aldehydes and ketones

20–50 mL Hydranal-Working Medium K or Hydranal-Medium K or Hydranal-KetoSolver are added to the titration vessel and titrated to a stable end point with Hydranal-Composite 5 K. The sample is then added and immediately titrated to a stable end point.

By using these reagents and following the recommended titration procedures, the side reactions of acetal or ketal formation and the bisulfite addition are significantly suppressed. Consequently, interferences are not encountered in the titration of aldehydes and ketones. Other techniques can also reduce the influence of these negative side reactions in certain cases, as described in the following discussion.

The bisulfite addition reaction begins upon addition of the sample to the sulfur-dioxide-containing working medium. Therefore, the KF titration should be started immediately to prevent any water present in the titration vessel from undergoing the bisulfite addition. We utilize the ‘flying start’ method whereby the sample is added within 20 seconds of the start of the titration. The instrument initiates the titration as soon as the sample is added. Titrators should be programmed to add the reagent rapidly for the same reason. However, commercially available instruments vary greatly in this respect.

Despite such precautions, some of the water can still be bound as bisulfite adduct, especially when titrating aromatic aldehydes. The dissociation of the bisulfite adduct must first occur in order to run a reliable determination of the water content in the sample. This is possible by using Hydranal-K reagents since they sufficiently suppress the formation of acetals and ketals.

The amount of water to be titrated should be low enough so that titrations are not inordinately long. We found sample sizes that contain a total of 10–25 mg H_2O are ideal. This amount of water consumes 2–5 mL reagent.

We have investigated the moisture determination of a number of aldehydes and ketones during the development of the Hydranal-K reagents. These compounds are listed in Table 9.6. The table shows the name of the chemical and the water content. The water content given is for reference only and is not to be taken as a limit. Column 3 lists the size of the sample that can be titrated in a 25 mL volume of Hydranal-Working Medium K or Hydranal-Medium K. The entry '10 mL' or '10 g' represents the largest sample size analyzed. Smaller sample sizes are indicated in column 4 with a designation for the reason of the limited sample size:

B = bisulfite addition
I = indication interferences
L = limited solubility
A = buffering of acid

The data in Table 9.6 shows that the determination of water in most ketones is straightforward. 10 mL samples of aliphatic ketones can be titrated without any interference, even with acetone and cyclohexanone, which are particularly reactive. However, trifluoroacetone gives a noticeable bisulfite addition reaction, so a 'flying start' titration and a verification of end point are required for its reliable determination.

Aromatic ketones and long-chain aliphatic ketones are less reactive and can also be titrated with Hydranal-Composite 5 and Hydranal-Medium K or Hydranal-Working Medium K or Hydranal-KetoSolver or Hydranal-CompoSolver E (the use of Hydranal-Composite 5 K is not necessary). Most heterocyclic ketones perform similarly to aromatic ketones. With acetyl pyridine the bisulfite addition is apparently activated by the pyridyl group, and therefore interferes with the water determination.

Diketones usually behave like normal ketones. Exceptions are diacetyl ketone and 1,2-cyclohexanedione to a certain extent. The adjacent keto groups, particularly in diacetyl ketone, are very reactive and only small amounts of sample can be analyzed. This is not the case with benzyl ketone presumably due to the aromatic substituents present in this compound.

Keto-carboxylic acids shift the pH of the working medium and delay the course of the titration. Buffering the working medium slightly accelerates the titration and restores the pH.

Procedure 9.6.1.2 Keto-carboxylic acids

25 mL Hydranal-Medium K or Hydranal-Working Medium K or Hydranal-KetoSolver or Hydranal-CompoSolver E are added to the titration vessel, mixed with 0.1–0.5 g Hydranal-Imidazole and titrated to dryness with Hydranal-Composite 5 K. The keto-carboxylic acid sample is then added and titrated in the usual manner.

Keto-carboxylic acids can be titrated according to procedure 9.6.1.2. Exceptions are 2-oxo-propionic acid and 2-oxobutyric acid (alpha-keto acids), which exhibit a strong tendency to undergo the bisulfite addition. The amounts of Hydranal-Imidazole added must be kept small since this reagent enhances the bisulfite addition.

The pH of the working medium is not shifted by the esters of keto-carboxylic acids, and they can be titrated according to procedure 9.6.1.1.

Many aldehydes can be analyzed in a similar manner. The formation of acetals cannot be detected under these titration conditions. On the other hand, the bisulfite addition takes place very rapidly and the sample sizes usually have to be reduced. The 'flying start' method is a good way of reducing the influence of the bisulfite addition.

Aromatic aldehydes are less reactive and consequently present fewer problems. Aliphatic aldehydes are more reactive. The formation of acetal with acetaldehyde is particularly strong, and a sample size of only 2 mL should be used for the titration. The reactivity decreases with increasing chain length and the sample size can be increased to 5 mL starting with butyraldehyde (see L 248).

Formaldehyde does not undergo acetal formation and can be titrated with methanolic reagents as in standard volumetric procedures. However, the total water content cannot be determined. Typically only 50% H_2O is found in a 35% formaldehyde solution. Part of the water is bound as paraformaldehyde. The total water content can be determined by carrying out the titration at 50°C. Details can be found in Laboratory Reports L 006 and L 386.

A glyoxal solution (40%) behaves similarly to formamide and can be titrated at elevated temperature (L 267). With a glutaraldehyde solution (50%) we titrated free water at room temperature and total water content at 50°C.

Table 9.6. Titration procedures for aldehydes and ketones.

SUBSTANCE	WATER CONTENT	TOTAL AMOUNT	RESTRICTION
Aliphatic ketones			
Acetone	0.064%	10 mL	
Methyl-n-propyl ketone	0.22%	10 mL	
Methyl-isobutyl ketone	0.041%	10 mL	
Ethyl-isobutyl ketone	0.39%	10 mL	
Allyl acetone	0.19%	10 mL	
3-Octanone	0.082%	10 mL	
2-Decanone	0.080%	10 mL	
Dihexyl-ketone	0.0086%	5 g	I
Cyclohexanone	0.032%	10 mL	
1,1,1-Trifluoroacetone	0.25%	10 mL	B
Hexachloroacetone	0.12%	5 mL	I
Aromatic ketones			
Acetophenone	0.029%	10 mL	
2-Fluoroacetophenone	0.21%	10 mL	
2,4-Dihydroxyacetophenone	0.021%	5 g	L
2-Aminoacetophenone	0.13%	10 mL	
Benzylmethyl ketone	0.038%	10 mL	
Benzylacetone	0.64%	10 mL	
Benzophenone	0.0032%	5 g	I
Benzoin	0.043%	2 g	L
Heterocyclic ketones			
2-Acetylpyridine	0.39%	10 mL	B
2-Pyrrolidone	0.058%	10 mL	
N-Methyl-2-pyrrolidone	0.021%	10 mL	
2-Benzoylpyridine	0.016%	10 g	
3-Acetylindol	0.34%	2 g	L
Diketones			
Diacetyl	0.10%	1 mL	B
Acetylacetone	0.043%	10 mL	
2,5-Hexandione	0.32%	10 mL	
1,2-Cyclohexanedione	0.90%	1 g	B
Benzoylacetone	0.037%	10 g	
Benzil (Dibenzoyl)	0.032%	10 g	
Dibenzoylmethane	0.036%	10 g	
Keto-carboxylic acids and derivates			
2-Oxo-propionic acid	1.07%	10 mL	B, A
2-Oxo-butyric acid	0.95%	1 g	B, A
Levulinic acid	0.22%	10 mL	A
3-Phenyl propionic acid	0.020%	5 g	L
2-Acetylbenzoic acid	0.079%	5 g	L, A
2-Benzoyl benzoic acid	0.94%	10 mL	
Ethyl acetoacetate	0.52%	10 mL	
Ethyl levulinate	0.057%	10 mL	
Ethyl benzoylacetate	0.033%	10 g	
Aliphatic aldehydes			
Acetaldehyde	0.021%	2 mL	B
Propionaldehyde	0.15%	2 mL	B
n-Butyraldehyde	0.035%	5 mL	B
Crotonaldehyde	0.10%	5 mL	B
Octaldehyde	0.26%	5 mL	B
Glycolaldehyde	0.25%	1 g	B, L
Chloral	0.12%	10 mL	exothermic
Chloral hydrate	10.86%	0.5 g	high water content
Bromal		0	I
Paraldehyde	0.018%	10 mL	
Cyclohexane carbaldehyde	0.027%	5 mL	
Diphenylacetaldehyde	0.11%	10 mL	B
Acetaldehyde diethylacetal	0.029%	10 mL	
Bromoacetaldehyde diethylacetal	0.043%	10 mL	I
Aromatic aldehydes			
Benzaldehyde	0.13%	5 mL	B
2-Bromobenzaldehyde	0.10%	2 mL	B
Salicylaldehyde	0.027%	10 mL	
3-Hydroxybenzaldehyde	0.22%	5 g	B
2-Anisaldehyde	0.040%	10 mL	B
4-Dimethylaminobenzaldehyde	0.016%	10 g	
Phenylglyoxal	1.00%	0.5 g	B

B = bisulfite addition, **I** = indication interferences, **L** = limited solubility, **A** = buffering of acid

9.6.2 Coulometric titration

We have also developed reagents for the coulometric determination of water in ketones:

- Hydranal-Coulomat AK
- Hydranal-Coulomat CG-K

Hydranal-Coulomat AK is the anolyte and is added to the anodic compartment of the titration cell. Hydranal-Coulomat CG K is the corresponding catholyte.

The solvent system of each reagent has been carefully made up to meet the demands of ketone analysis using modern KF instruments. The composition of these reagents has been optimized and should not be altered by the addition of other solvents. For the same reason, no more than 20 mL of liquid sample per 100 mL of the anolyte reagent should be used. The same restrictions apply to the analysis of solids dissolved in solvents. We recommend a 4:1 (v/v) solution of 2-methoxyethanol and Hydranal-Chloroform, or the solvents used individually, because they ensure minimal alteration to the electrolytic properties of the anolyte.

Use of methanol as the solvent is particularly detrimental because it enhances the formation of ketals. The coulometric titration cell must be thoroughly cleaned when replacing conventional methanol-containing coulometric reagents with Hydranal K-type reagents. If ketones are analyzed on a regular basis, we recommend having a separate coulometric titration cell dedicated to this analysis to prevent the need for frequent cleaning or the possibility of methanol contamination.

Iodine solutions based on methanol must not be used to dry the reagents used in the ketone titration cell. We recommend the use of Hydranal-Composite 5 or a solution of iodine in diethylene glycol monoethyl ether.

The K-type reagents can be used in the usual way for the determination of water in ketones. The samples sizes should be relatively small, preferably 1 mL. The sample size of reactive ketones, such as cyclohexanone, should only be 0.2 mL or 0.5 mL. Larger samples can cause serious instrument drift and eventually an end point will not be reached. The instrument also influences the sample size.

After several ketone samples have been analyzed in the K-type reagent, the instrument indicates a drift or a residual current. This drift corresponds to the amount of water that the instrument removes per minute. This also means that in a drifting cell there is a continual consumption of reagent. It is therefore understandable that a titration cell that has been used for a number of successive ketone titrations will have a permanent consumption of reagent. The reagent in the cell will be spent within a few days even if it has not been used for the titration of further samples.

Aldehydes can be analyzed with the same reagents but with some restrictions. Aldehydes undergo the same side reactions, but more rapidly than corresponding ketones. The water content of benzaldehyde, representative of aromatic aldehydes, can be determined with an acceptable degree of accuracy if the sample size is restricted to 0.5 mL. Aromatic aldehydes undergo the bisulfite addition and, like ketones, the dissociation of the bisulfite adduct must occur first in order to run a reliable determination of the water content in the sample. The acetal formation with n-butyraldehyde is particularly strong and the delay time of the instrument should not be set too high. This side reaction decreases with increasing chain length. Side reactions predominate in acetaldehyde to such an extent that it cannot be analyzed.

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An accurate determination of water in aldehydes should be carried out using the volumetric titration with Hydranal-Composite 5 K and Hydranal-Medium K or Hydranal-Working Medium K or Hydranal-KetoSolver. Hydranal-Coulomat AK and Hydranal-Coulomat CG-K can also be used to investigate other compounds, such as hydrocarbons, halogenated hydrocarbons, or alcohols. They are not suitable for the analysis of acids and bases.

Using the same reagent for the determination of the water content of a mixture of ketones and other substances is possible if the substance does not chemically react with the ketone. Therefore, alcohols cannot be investigated in the presence of ketones.

We have found it economical and practical to titrate aldehydes/ketones and other compounds in separate, dedicated cells. The standard reagents for coulometry, Hydranal-Coulomat A/AG/AG-H/E and Hydranal-Coulomat CG, have a significantly higher water capacity than the Hydranal-K reagents.

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Water determination in insulating oil

ESSO UNIVOLT 50 and 56, paraffinic uninhibited transformer oils

HYDRANAL™ Laboratory Report L 462

A coulometric determination was conducted because of the low water content. Since purely alcoholic reagents are not suitable for extracting water with multiple injections, we used reagents that contain solubilizers. While very few oil samples clearly dissolved, they did disperse. Using reagents that contain chloroform and/or xylene requires the use of a coulometry cell with diaphragm.

We found 35 ppm of water with direct injection.

We also followed the indirect method with a KF oven for vials. The analyses showed that 80°C was enough to release the water. We chose to precisely weigh in 4 mL oil sample and we found 26 ppm of water.

In this procedure, the carrier gas is fed directly into the sample. We noted that some 25% of the sample had transferred to the coulometry cell by the end of the determination, because the sample was frothing.

The sample was reduced to around 2 mL so that the carrier gas no longer passed through the sample. This suppressed the frothing. As compensation, we set the minimum determination time to 900 seconds. We found 24 ppm of water.

Procedure for direct coulometric titration in cell with diaphragm:

Add approx. 100 mL Hydranal-Coulomat Oil or Hydranal-Coulomat A to the anodic compartment of a coulometric cell with a diaphragm and then add Hydranal-Coulomat CG to the cathodic compartment to the same level (usually 5 mL).

The machine is switched on and it titrates automatically to dryness. When the instrument is showing a low, stable drift, samples can be weighed in by difference using a syringe.

Hydranal-Water Standard 1.0 and Hydranal-Water Standard 0.1 PC are suitable for controlling the coulometric cell.

Procedure for indirect coulometric titration with KF oven:

Place 150 mL Hydranal-Coulomat AG-Oven in the anode compartment of a coulometric cell with diaphragm. Fill the cathode compartment to the same level with Hydranal-Coulomat CG. The cell without diaphragm only requires 150 mL of Hydranal-Coulomat AG-Oven.

The machine is switched on and automatically titrates to dryness. When the drift is low and stable, the carrier gas is connected. When the original stable drift value is approximately reached with the carrier gas, the sample can be weighed in precisely by means of differential weighing and heated to 80°C.

The blank value of the method must be determined and be included in the calculations.

Hydranal-Molecular Sieve 0.3 nm or Hydranal-Humidity Absorber is very well suited as a drying agent for the carrier gas.

Hydranal-Water Standard 1.0 and Hydranal-Water Standard 0.1 PC are suitable for controlling the coulometric cell. Hydranal-Water Standard KF Oven 140–160°C is suitable for controlling the KF oven.

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REAGENTS

34805	HYDRANAL-Composite 5	34801	HYDRANAL-Titrant 5
34741	HYDRANAL-Methanol dry	34800	HYDRANAL-Solvent
37817	HYDRANAL-Methanol Rapid	34836	HYDRANAL-Coulomat AG
37859	HYDRANAL-Buffer Base	34840	34840 HYDRANAL-Coulomat CG
32035	HYDRANAL-Benzoic acid	34820	HYDRANAL-Coulomat AK
37865	HYDRANAL-Salicylic acid	34821	HYDRANAL-Coulomat CG-K

WATER STANDARDS

34849	HYDRANAL-Water Standard 10.0	34426	HYDRANAL-CRM Water Standard 1.0
34425	HYDRANAL-CRM Water Standard 10.0	34446	HYDRANAL-Water Standard 0.1 PC
34828	HYDRANAL-Water Standard 1.0		

AUXILIARIES

34241	HYDRANAL-Molecular Sieve 0.3 nm	34788	HYDRANAL-Humidity Absorber

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Water determination in crude oil

HYDRANAL™ Laboratory Report L 108

From the analysis of many different oils, we have learned that intensive homogenization of samples using a homogenizer or by ultrasound is a fundamental prerequisite for reproducible results.

Six different crude oil samples that we analyzed had water contents of between 0.05% and 0.32%, and for sample oil the same result was obtained using both analytical methods.

Crude oil requires different solvents to aid solubility: chloroform to dissolve the oil and xylene to dissolve the tar components.If the tar is not finely dispersed, it can coat the electrode, which leads to indication problems.

According to ASTM D 4377-00, in analysis using a pyridine-free reagent, a mixture of a Karl Fischer solvent (for example Hydranal-Solvent) and xylene must be added to the titration vessel. Hydranal-Solver (Crude) Oil fulfills all these requirements.

Procedure for volumetric one-component titration:

Add 30 mL Hydranal-Solver (Crude) Oil to the titration vessel and titrate to dryness using Hydranal-Composite 5. Accurately weigh-in by difference approximately 4 g sample and titrate the water content using Hydranal-Composite 5.

Hydranal-Water Standard 10.0, Hydranal-Water Standard 1.0, and Hydranal-Standard Sodium Tartrate Dihydrate are suitable for determination of the titer or control of the volumetric determination.

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Procedure for coulometric titration:

Add approx. 100 mL of Hydranal-Coulomat Oil to the anode compartment of a coulometric cell with a diaphragm and then add Hydranal-Coulomat CG to the cathode compartment to the same level (usually 5 mL).

Instead of Hydranal-Coulomat Oil, a mixture of 70 mL Hydranal-Coulomat A and 30 mL Hydranal-Xylene could also be used.

The machine is switched on and it titrates automatically to dryness. When the instrument is showing a low, stable drift, samples can be weighed in by difference using a syringe.

Hydranal-Water Standard 1.0 and Hydranal-Water Standard 0.1 PC are suitable for controlling the coulometric cell.

The coulometric method is cumulative. After the first determination, the solution becomes dark and visual control is not possible. We have, however, found that results from one oil source are reproducible and that there are therefore no indication problems for the analysis. During our tests, we injected a total of approx. 30 g crude oil into the cell. Each individual analysis was of between 1 and 2 g oil, depending on the water content of the sample.

VOLUMETRIC REAGENTS

34805 HYDRANAL-Composite 5
34697 HYDRANAL-Solver (Crude) Oil
34849 HYDRANAL-Water Standard 10.0
34425 HYDRANAL-CRM Water Standard 10.0
34828 HYDRANAL-Water Standard 1.0
34426 HYDRANAL-CRM Water Standard 1.0
34696 HYDRANAL-Standard Sodium Tartrate Dihydrate
34424 HYDRANAL-CRM Sodium Tartrate Dihydrate

COULOMETRIC REAGENTS

34868 HYDRANAL-Coulomat Oil
34807 HYDRANAL-Coulomat A
37866 HYDRANAL-Xylene
34840 HYDRANAL-Coulomat CG
34828 HYDRANAL-Water Standard 1.0
34426 HYDRANAL-CRM Water Standard 1.0
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