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Hydranal™ Honeywell Consistency Innovation

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HYDRANAL™ Product Overview Guide
Reagents for water determination by Karl Fischer titration

Honeywell

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by Thermo Fisher Scientific

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:



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 or coulometrically.



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.

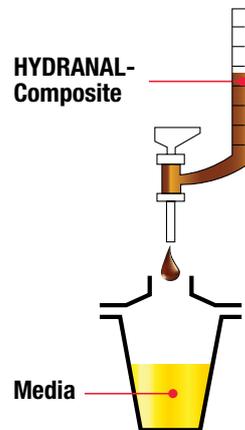
Volumetric One-Component Titration - Titrating Agents

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.

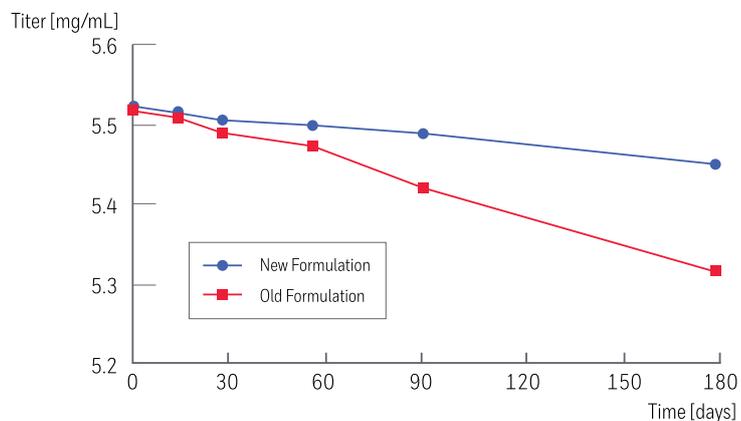


Figure 1. Results of the titer stability tests

FS Part Number	HON Product Number	Product Name	Description
6002563	34805-1LUS	HYDRANAL-Composite 5	One-component reagent, titer ~5 mg/mL
6002564	34805-2.5LUS	HYDRANAL-Composite 5	One-component reagent, titer ~5 mg/mL
6002565	34805-500MLUS	HYDRANAL-Composite 5	One-component reagent, titer ~5 mg/mL
6002566	34806-1L	HYDRANAL-Composite 2	One-component reagent, titer ~2 mg/mL
6002567	34806-2.5LUS	HYDRANAL-Composite 2	One-component reagent, titer ~2 mg/mL
6002568	34806-500MLUS	HYDRANAL-Composite 2	One-component reagent, titer ~2 mg/mL
6002569	34816-1LUS	HYDRANAL-Composite 5 K	One-component reagent for titration of ketones and aldehydes, titer ~5 mg/mL
6002570	34816-2.5LUS	HYDRANAL-Composite 5 K	One-component reagent for titration of ketones and aldehydes, titer ~5 mg/mL
6002571	34816-500MLUS	HYDRANAL-Composite 5 K	One-component reagent for titration of ketones and aldehydes, titer ~5 mg/mL
6002573	34827-1LUS	HYDRANAL-Composite 1	One-component reagent, titer ~1 mg/mL
6002574	34827-500MLUS	HYDRANAL-Composite 1	One-component reagent, titer ~1 mg/mL

Volumetric One-Component Titration - Media

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

HYDRANAL-Methanol Rapid

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.

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).

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.

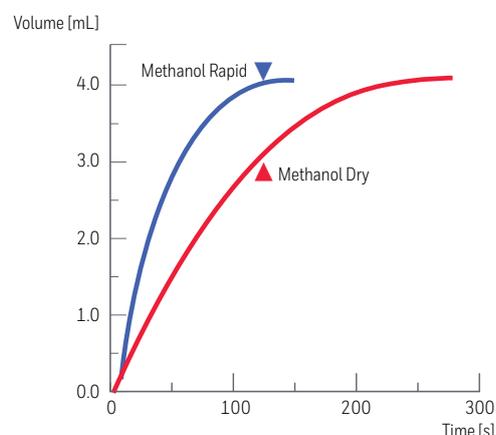


Figure 2. Titration of 20 mg water

Advantages of HYDRANAL-Methanol Rapid:

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

FS Part Number	HON Product Number	Product Name	Description
6000672	34697-1L	HYDRANAL-Solver (Crude) Oil	Working medium containing methanol, xylene and chloroform for titration in oils
6000673	34697-2.5L	HYDRANAL-Solver (Crude) Oil	Working medium containing methanol, xylene and chloroform for titration in oils
6000658	34698-1LUS	HYDRANAL-Medium K	Less toxic working medium containing chloroform for ketones and aldehydes
6000640	34734-1L	HYDRANAL-CompoSolver E	Ethanol-based medium containing accelerators
6000641	34734-2.5L	HYDRANAL-CompoSolver E	Ethanol-based medium containing accelerators
6000654	34738-1LUS	HYDRANAL-KetoSolver	Working medium free of halogenated solvents for ketones and aldehydes
6000655	34738-500ML	HYDRANAL-KetoSolver	Working medium free of halogenated solvents for ketones and aldehydes
6000659	34741-1LUS	HYDRANAL-Methanol Dry	Medium for general use
6000660	34741-2.5LUS	HYDRANAL-Methanol Dry	Medium for general use
6000684	34817-1L	HYDRANAL-Working Medium K	Working medium containing chloroform and 2-chloroethanol for ketones and aldehydes
6000661	37817-1LUS	HYDRANAL-Methanol Rapid	Medium containing accelerators
6000662	37817-2.5LUS	HYDRANAL-Methanol Rapid	Medium containing accelerators
6000656	37855-1L	HYDRANAL-LipoSolver CM	Working medium containing methanol and chloroform for titration in non-polar samples
6000657	37856-1L	HYDRANAL-LipoSolver MH	Working medium containing methanol and 1-hexanol for titration in non-polar samples

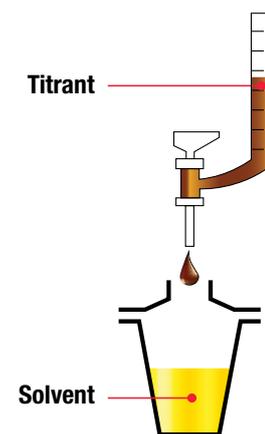
Volumetric Two-Component Titration

HYDRANAL-Titrant / Solvent

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.



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)

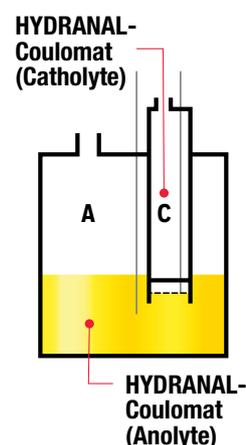
FS Part Number	HON Product Number	Product Name	Description
Methanol Based			
6003223	34811-1LUS	HYDRANAL-Titrant 2	Two-component reagent, titer ~2 mg/mL
6003224	34811-2.5L	HYDRANAL-Titrant 2	Two-component reagent, titer ~2 mg/mL
6003225	34811-500MLUS	HYDRANAL-Titrant 2	Two-component reagent, titer ~2 mg/mL
6003226	34801-1LUS	HYDRANAL-Titrant 5	Two-component reagent, titer ~5 mg/mL
6003227	34801-2.5L	HYDRANAL-Titrant 5	Two-component reagent, titer ~5 mg/mL
6003228	34801-500ML	HYDRANAL-Titrant 5	Two-component reagent, titer ~5 mg/mL
6000670	34800-1LUS	HYDRANAL-Solvent	Working medium for two-component titration
6000671	34800-2.5LUS	HYDRANAL-Solvent	Working medium for two-component titration
Ethanol Based			
6000676	34723-1LUS	HYDRANAL-Titrant 2 E	Two-component reagent, titer ~2 mg/mL
6000678	34732-1LUS	HYDRANAL-Titrant 5 E	Two-component reagent, titer ~5 mg/mL
6000679	34732-2.5LUS	HYDRANAL-Titrant 5 E	Two-component reagent, titer ~5 mg/mL
6000680	34732-500ML	HYDRANAL-Titrant 5 E	Two-component reagent, titer ~5 mg/mL
6000666	34730-1L	HYDRANAL-Solvent E	Working medium for two-component titration
6000667	34730-2.5L	HYDRANAL-Solvent E	Working medium for two-component titration
6000668	34730-500ML	HYDRANAL-Solvent E	Working medium for two-component titration
Special Media			
6000664	34812-1LUS	HYDRANAL-Solvent CM	Working medium for two-component titration, containing methanol and chloroform for titration in non-polar samples
6000665	34812-2.5LUS	HYDRANAL-Solvent CM	Working medium for two-component titration, containing methanol and chloroform for titration in non-polar samples
6000669	34749-1L	HYDRANAL-Solvent Oil	Working medium for two-component titration, containing methanol and 1-hexanol for titration in non-polar samples
6000672	34697-1L	HYDRANAL-Solvent (Crude) Oil	Working medium containing methanol, xylene and chloroform for titration in oils
6000673	34697-2.5L	HYDRANAL-Solvent (Crude) Oil	Working medium containing methanol, xylene and chloroform for titration in oils

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. 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 a trace range of water).

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)

FS Part Number	HON Product Number	Product Name	Description
60046449	34807-500MLUS	HYDRANAL-Coulomat A	Anolyte preferred for cells with diaphragm*
6000645	34836-500MLUS	HYDRANAL-Coulomat AG	Anolyte suitable for cells with and without diaphragm
6000644	34836-1LUS	HYDRANAL-Coulomat AG	Anolyte suitable for cells with and without diaphragm
6000646	34843-500MLUS	HYDRANAL-Coulomat AG-H	Anolyte for titration of long-chained hydrocarbons, preferred for cells with diaphragm*
6000647	34739-500MLUS	HYDRANAL-Coulomat AG-Oven	Anolyte for determination with Karl Fischer oven, suitable for cells with and without diaphragm
6000648	34820-500MLUS	HYDRANAL-Coulomat AK	Anolyte for titration of ketones, preferred for cells with diaphragm*
6000651	34868-100ML	HYDRANAL-Coulomat Oil	Anolyte for titration of oils, preferred for cells with diaphragm*
6000652	34868-500MLUS	HYDRANAL-Coulomat Oil	Anolyte for titration of oils, preferred for cells with diaphragm*
6000650	34726-500ML	HYDRANAL-Coulomat E	Anolyte based on ethanol, suitable for cells with and without diaphragm
6000642	34810-500MLUS	HYDRANAL-Coulomat AD	Anolyte preferred for cells without diaphragm
6000649	34840-50ML	HYDRANAL-Coulomat CG	Catholyte
6002572	34821-50ML	HYDRANAL-Coulomat CG-K	Catholyte for titration of ketones

* 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 the NIST (National Institute of Standards and Technology, USA) standard reference material SRM 2890, Water Saturated Octanol.

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.

FS Part Number	HON Product Number	Product Name	Description
60050001	34425-80ML	HYDRANAL-CRM Water Standard 10.0	Liquid CRM standard, water content 10.0 mg/g = 1.0%
60050002	34426-40ML	HYDRANAL-CRM Water Standard 1.0	Liquid CRM standard, water content 1.0 mg/g = 0.1%
60050000	34424-10G	HYDRANAL-CRM Sodium Tartrate Dihydrate	Solid CRM standard, water content ~15.66%
6000683	34849-80ML	HYDRANAL-Water Standard 10.0	Liquid standard, water content 10.0 mg/g = 1.0%
6000682	34828-40ML	HYDRANAL-Water Standard 1.0	Liquid standard, water content 1.0 mg/g = 0.1%
6000681	34847-40ML	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)
60050003	34446-40ML	HYDRANAL-Water Standard 0.1 PC	Liquid standard water content 0.1 mg/g = 0.01% (improved stability compared to 34847: shelf life 3 years, to be stored at room temp.)
6002550	34694-80ML	HYDRANAL-Water Standard Oil	Liquid standard based on mineral oil, water content <50 ppm (0.005%)
6002551	34696-25G	HYDRANAL-Standard Sodium Tartrate Dihydrate	Solid standard, water content ~15.66%
6002549	34693-10G	HYDRANAL-Water Standard KF Oven 140-160°C	Solid standard for control of Karl Fischer ovens, water content ~5%, based on lactose
6002553	34748-10G	HYDRANAL-Water Standard KF Oven 220-230°C	Solid standard for control of Karl Fischer ovens, water content ~5.55%, based on potassium citrate

Auxiliaries for Karl Fischer Titration

Solubilizers

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

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.

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.

FS Part Number	HON Product Number	Product Name	Description
6002552	34724-1L	HYDRANAL-Formamide Dry	Solubilizer, max. 0.02% water
6000639	37863-1L	HYDRANAL-Chloroform	Solubilizer, max. 0.01% water
6000685	37866-1L	HYDRANAL-Xylene	Solubilizer, max. 0.01% water
6000637	34804-500MLUS	HYDRANAL-Buffer Acid	Liquid buffer medium, based on imidazole
6000638	37859-1L	HYDRANAL-Buffer Base	Liquid buffer medium, based on salicylic acid
6002401	32035-500G	HYDRANAL-Benzoic Acid	Buffer substance
6002654	37865-500G	HYDRANAL-Salicylic Acid	Buffer substance
6000653	37864-500G	HYDRANAL-Imidazole	Buffer substance
6000674	34813-100ML	HYDRANAL-Standard 5.0	Test solution for volumetric titration, water content 5.00 mg/mL
6000675	34813-500ML	HYDRANAL-Standard 5.0	Test solution for volumetric titration, water content 5.00 mg/mL
6002560	34803-100G	HYDRANAL-Sodium Tartrate Dihydrate	Test substance for volumetric titration, water content ~15.66%
6000973	34802-1L	HYDRANAL-Water-in-Methanol 5.0	Reagent for volumetric back titration, water content 5.00 mg/mL
6000974	34802-500ML	HYDRANAL-Water-in-Methanol 5.0	Reagent for volumetric back titration, water content 5.00 mg/mL
6002558	34788-1KG	HYDRANAL-Humidity Absorber	Drying agent for air and gases with indicator
6002559	34788-500G	HYDRANAL-Humidity Absorber	Drying agent for air and gases with indicator
6002483	34241-250G	HYDRANAL-Molecular Sieve 0.3 nm	Drying agent for air and gases
6000663	37858-1EA	HYDRANAL-Moisture Test Kit	Test kit for the visual water determination according to Karl Fischer without titrator

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

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