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HYDRANAL™ Technical Information Sheet T004

Titer Determination of Hydranal-Composite One-Component Reagents in Alcoholic Media.
Comparison: Liquid Hydranal-Water Standard 10.0 vs. solid Hydranal-Standard Sodium Tartrate Dihydrate

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

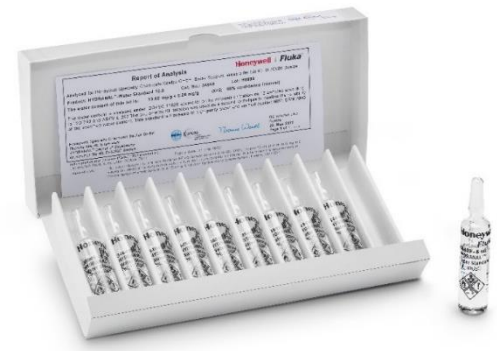

Fig. 1. Hydranal-Water Standard 10.0.

Liquid standards, like Hydranal-Water Standard 10.0 (water content 10 mg/g = 1%), 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).

Fig. 2. Hydranal-Standard Sodium Tartrate Dihydrate.

Solid standards, like Hydranal-Standard Sodium Tartrate Dihydrate (water content ~15.66%), contain defined amounts of chemically bound water. They are suitable for both general use in volumetry as well as for the Karl Fischer oven. These standards are packed in amber glass bottles.

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Liquid vs. solid Water Standards

Historically, sodium tartrate dihydrate has been used as a reliable water standard due its defined amount of crystallization water.
Nowadays, more and more laboratories are switching to a new source of water by using innovative liquid water standards which provide pure water diluted with inert solvent systems. Both solid and liquid standards can be used for titer determination. However, there are some benefits and drawbacks. Results of our comparison tests are described in Table 1.

Table 1. Titer determination of Hydranal-Composite 5 in Hydranal-Methanol Rapid with solid and liquid water standard, number of determinations n = 5. Testing conditions: temperature 23°C; fast stirring speed; large stirring bar.

	Hydranal-Standard Sodium Tartrate Dihydrate	Hydranal-Water Standard 10.0
Water content [mg/g]	156.6	10.0
Solubility	Moderate	Endless
Use of solvent	5 x 50 mL	1 x 30 mL
Sum of used solvent	250 mL	30 mL
Sample size	100 mg	1.5 mL
Mean Titer [mg/mL]	5.3427	5.3433
S(abs) [mg/mL]	0.00740	0.00163
S(rel) [%]	0.14	0.03
Min Titer [mg/mL]	5.3356	5.3407
Max Titer [mg/mL]	5.3536	5.3451
Mean titration time	130 s	82 s

Summary

Hydranal-Water Standard 10.0 has an unlimited solubility in all solvents. Only 30 mL of Hydranal-Methanol Rapid are required for a fivefold determination.

Due to the limited solubility of sodium tartrate dihydrate in methanol, the solvent must be replaced after each titration. Therefore, the titer determination with sodium tartrate dihydrate requires at least eight times more solvent.

Both standards provide a titer of 5.343 mg/mL. Whereby, the titer determination with the liquid Hydranal-Water Standard 10.0 shows a better standard deviation and higher accuracy. Additionally, the process of using liquid standard is significantly faster due to shorter titration time (Fig. 3) and no requirement of multiple solvent changes

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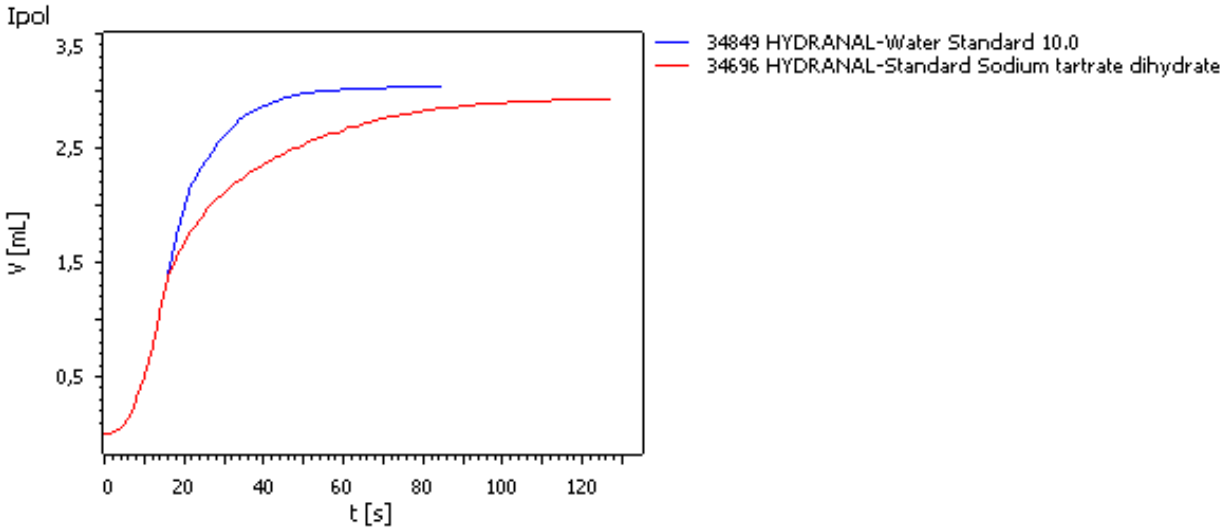

Fig. 3. Titration curves for Hydranal-Water Standard 10.0 and Hydranal-Standard Sodium Tartrate Dihydrate in Hydranal-Methanol Rapid.

To learn more about accurate titer determination and the influence of e.g. decomposition or environment on the titer, read HYDRANAL™ Technical Information Sheet T001 “Determining the titer of Karl Fischer reagents and influences on accuracy”.

Volumetric reagents:
34805 HYDRANAL-Composite 5
37817 HYDRANAL-Methanol Rapid

Water Standards:
34849 HYDRANAL-Water Standard 10.0
34425 HYDRANAL-CRM Water Standard 10.0
34696 HYDRANAL-Standard Sodium Tartrate Dihydrate
34424 HYDRANAL-CRM Sodium Tartrate Dihydrate

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