APPLICATION REPORT

Keeping the Good In

Potassium Bitartrate Stability after Sheet Filtration sheets

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Wine filtration using depth filter sheets in plate and frame filters is a well-known enological process, which, although well established, is continuously under development. The depth filter sheet is used to separate haze and microorganisms such as yeast or malolactic bacteria in a reliable manner, while retaining valuable contents such as aroma and color, and thus a wine's typicity. There is a large selection of depth filter sheets available to winemakers, which differ in composition, matrix and adsorption capacity (zeta potential). Choosing the right depth filter sheet depends on the relevant filtration requirements.

For instance, bottled wines must be stable and not contain any crystal precipitations (potassium bitartrate). This visual defect can lead to consumer complaints. In order to combat potassium bitartrate precipitations, additional colloids, such as metatartaric acid, are added. These bind to the active centers of the crystal surfaces (potassium hydrogen tartrate), thereby preventing crystal formation due to their multiple molecule layers. This stabilizes the wine's colloidal structure. As a "protective colloid," the metatartaric acid prevents the crystallization of potassium bitartrate in wines.

However, stabilizing potassium bitartrate using metatartaric acid presents an additional challenge for depth filter sheet filtration. Just like the valuable components such as aroma and color, the colloidally dissolved metatartaric acid must pass through the depth filter sheet, i.e. it must not be adsorbed.

As early as 1984, Professor Dr. Karl Wucherpfennig and Professor Dr. Helmut Dietrich were investigating the filtration behavior of CMC-stabilized wine (in Die Weinwirtschaft Technik, No. 12, 12/14/1984, pp. 350–353). They established that dissolved colloidal substances, such as metatartaric acid, have an effect on the performance of filter media: depending on the type and amount, they reduce filter performance. In their experiments with metatartaric-acid-stabilized wines and various filter sheets, they discovered that some colloidally dissolved substances were being filtered by filter sheets with a high adsorption capacity.

Metatartaric acid is a natural, high-molecular-weight and polymerized tartaric acid. It is created through the esterification of tartaric acid molecules and simultaneous dehydration. The effect of metatartaric acid is time-limited. Depending on the degree of esterification (between 38% and 42%) and storage temperature, it naturally decomposes to tartaric acid and becomes inactive.

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The added protective colloid is therefore filtered out again from the first few liters of wine. This quantity of filtered wine, therefore, does not achieve potassium bitartrate stability, which is not ideal. The goal is to achieve potassium bitartrate stability in the whole of the filtrate from the outset.

Materials and Methods

In order to investigate this phenomenon more closely and identify suitable filtration solutions, Eaton's Filtration Division tested various filter media. Various BECOPAD® sterile filter sheets made of high-purity cellulose fibers (C sterile filter sheet), sterile filter sheets containing perlite and diatomaceous earth (standard sterile filter sheets), and a pre-filter cartridge material made of polypropylene (BECO PROTECT® FS) were used.

The experiments were conducted by Benjamin Petry as part of his master's thesis (Hochschule Geisenheim University) in the technical lab of Eaton Technologies GmbH. The tests were conducted four times on the filtration stand (14 cm filter area) with a 2013 red wine made from the Regent grape. The filtrate was tested for its potassium bitartrate stability using filtration amounts of between 8 and 77 l/m² and was defined as having achieved potassium bitartrate stability at ≤ 40 µS. (the lower this guide value, the less potassium bitartrate crystallized).

The tests were carried out using two filtration techniques on a pilot-plant scale (approx. 500 liters): in the first test, the wine was filtered directly after the metatartaric acid was added; in the second test, filtration occurred after the metatartaric acid was allowed to react for 72 hours.

Filter medium	Filtrate (l/m²)									
	8	15	23	31	38	46	54	62	69	77
Control: unfiltered red wine, stabilized with metatartaric acid										
C sterile filter sheet 1										
C sterile filter sheet 2										
C sterile filter sheet 3	54 µS									
C sterile filter sheet 4										
Standard sterile filter sheet 1	169 µS									
Standard sterile filter sheet 2	356 µS	340 µS	270 µS	255 µS	204 µS	177 µS	143 µS	91 µS		
Pre-filter cartridge										

Key:
Potassium bitartrate stability achieved (≤ 40 µS); Potassium bitartrate stability not achieved (≥ 40 µS);

Table 1: Wine stability results: direct addition of metatartaric acid to wine with subsequent filtration

Results

The red wine that was filtered using the C sterile filter sheets in the first test (see Table 1) achieved potassium bitartrate stability after only 8 l/m² (excluding C sterile filter sheet 3, where stability was reached after 15 l/m²).

The results for the standard sterile filter sheets were extremely varied. Standard filter sheet 1 has the same adsorption capacity as C sterile filter sheet 3, such that the wine achieved potassium bitartrate stability after 15 l/m² in this case too. In contrast, the red wine filtered using standard sterile filter sheet 2 only achieved potassium bitartrate stability after 62 l/m².

The pre-filter cartridge material showed no adsorptive effect and so the wine achieved potassium bitartrate stability.

The second test showed that potassium bitartrate stability is negatively influenced by the 72-hour reaction time (see Table 2). Excluding C sterile filter sheet 3 and the pre-filter cartridge material, with which the filtrate achieves immediate potassium bitartrate stability, all other filtrate samples did not achieve potassium bitartrate stability after 8 l/m². In further samples after 15 l/m², only C sterile filter sheet 2 and standard sterile filter sheets 1 and 2 did not achieve potassium bitartrate stability and wine stability was only achieved after 23 l/m².

Filter medium	Filtrate (l/m²)									
	8	15	23	31	38	46	54	62	69	77
Control: unfiltered red wine, stabilized with metatartaric acid										
C sterile filter sheet 1	45 µS									
C sterile filter sheet 2	44 µS	42 µS								
C sterile filter sheet 3										
C sterile filter sheet 4	42 µS									
Standard sterile filter sheet 1	104 µS	41 µS								
Standard sterile filter sheet 2	350 µS	335 µS	245 µS	200 µS	164 µS	131 µS	80 µS	56 µS	45 µS	
Pre-filter cartridge										

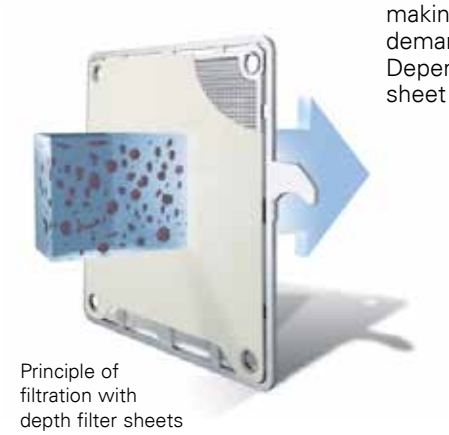
Key:
Potassium bitartrate stability achieved (≤ 40 µS); Potassium bitartrate stability not achieved (≥ 40 µS);

Table 2: Wine stability results: 72-hour metatartaric acid reaction time in the wine with subsequent filtration

Particularly striking are the test results for standard sterile filter sheet 2, with which the filtrate only achieved potassium bitartrate stability after 77 l/m².

In addition, the multi-factor analysis of variance showed that there is a significant difference ($P < 0.001$, corresponding to a 95% probability) between the three parameters of (1) the filter material used, (2) reaction time and (3) filtration volume.

The extrapolation for the C sterile filter sheets shows a significantly lower adsorptive effect, or no adsorptive effect, in comparison to the standard sterile filter sheets, regardless of the metatartaric acid reaction time. As per the analysis of variance, the filtrate regains potassium bitartrate stability after 8 l/m². Regarding standard sterile filter sheets, wine stability is heavily dependent on the formulation and contents (diatomaceous earth and perlite). Significant differences in adsorptive effect were calculated. Standard sterile filter sheet 2 showed a particularly high adsorptive potential.



Principle of filtration with depth filter sheets

Summary

The statement by Wucherpfennig und Dietrich that colloidally dissolved substances are adsorbed by filter media is attested by the experimental results for standard sterile filter sheet 2. It demonstrated a high level of binding of colloidally dissolved metatartaric acid (irrespective of the reaction time) on the filter sheet material. This adsorption capacity means that, in practice, the filtrate must be pumped back into the filtration loop until the adsorption capacity of the filter sheet is saturated and potassium bitartrate stability is achieved. This additional step is cost- and time-intensive and repeated filtration diminishes the wine's valuable components, such as aroma, color, and thus also typicity.

Wucherpfennig und Dietrich's results could not be applied to the BECOPAD depth filter sheet because, at the time of the experiment, there were no cellulose filter sheets available. Progress in product development towards depth filter sheets made of high-purity cellulose fibers means adsorptive behavior is reduced, making them suited to the demands of modern enology. Depending on the chosen filter sheet type, the retention rate and reaction time of metatartaric acid in wine, potassium bitartrate stability is achieved in the first liter of filtrate. In practice, these means easy handling and an optimal filtration result, while retaining maximum wine quality.

The tested pre-filter cartridge material is unrivaled. This filter material demonstrated no adsorptive properties, with the colloidally dissolved substances being retained from the start of filtration, irrespective of the metatartaric acid reaction time. This material is used exclusively in pre-filter cartridges. In practice, it cannot simply be used automatically; it requires a technological change from sterile filter sheets to a combination of pre-filter and membrane filter cartridges.

What do these new results mean for wine makers? Using a combination of metatartaric acid and sterile filter sheets made of high-purity cellulose, wine makers can bottle wine directly from the sheet filter without having to worry about visual defects such as potassium bitartrate precipitation. This also ensures that all of the wine's valuable components and its typicity are safely preserved in the bottle.

Depth filter sheets in various formats (BECOPAD®)

Sheet filter filtration system for 40 x 40 cm filter sheets (BECOCOMPACT® PLATE 400)

References:

WUCHERPFENNIG, K. AND DIETRICH, H., Filtrationsverhalten CMC-stabilisierter Weine [Filtration Behavior in CMC-stabilized Wines] in Die Weinwirtschaft Technik, No. 12, 12/14/1984, pp. 350–353

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