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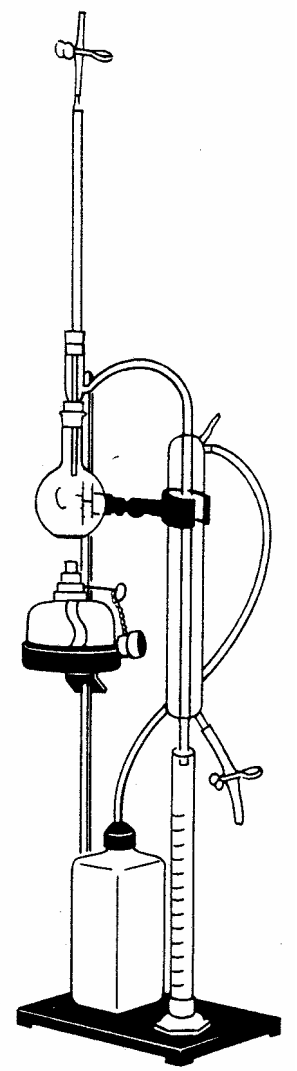
Getränkeanalytik

Determination of volatile acid according to the Wädenswil method

version 10/2005

Technical information and instructions for use



Working equipment for the determination of volatile acid according to the Wädenswil method:

- Stand with aluminium rod 600 x 12 mm
- 1 alcohol burner with tool holder
- 1 Wädenswil volatile acid distillation tube with water cooler (at the distillation tube silicone plug 21/16 x 25 mm, at cooler 2 plugs for water and 1 vent plug, at the lower water plug approx. 100 mm of silicone hose 5 x 1.5 mm and pinchcock according to Mohr, at the upper water plug approx. 500 mm of silicone hose 5 x 1.5 mm with screw cap for 500 ml poly bottle)
- 2 x 500-ml poly bottles
- 1 round clamp with sleeve, 40 mm span
- 2 distillation flasks (flat-bottomed flask100 ml NS 19 with heat protection)
- 1 graduated Wädenswil volatile acid pipette 7.5 : 2.5 ml with silicone plug 18/14 x 20 mm and approx. 60 mm of silicone hose 4 x 2 mm with TTS valve
- 2 graduated Wädenswil volatile-acid measuring cylinders with poly base
- 1 volumetric pipette 5 ml for the test sample
- 1 volumetric pipette 10 ml for preparing the 1/100 n caustic soda lye
- 1 volumetric flask 100 ml with poly plug for the same purpose
- 1 pack of pumice stones
- 1 spoon for pumice stones
- 1 bottle of silicone antifoam solution

Required reagents (not included in the price):

- 100 ml 1/100 n caustic soda lye (bottle with dosing cap)
- 100 ml 1/10 n caustic soda lye
- 50 ml indicator solution in dropping bottle

Analysis instructions:

- Fill cooler before each determination from the supply bottle connected to the upper liquid plug with cold water (collect after each determination the preheated water at the lower plug in a second bottle by using the pinchcock) or cool it continuously in case of a corresponding test capacity (in this case the cooling water is fed through the lower plug and discharged through the upper plug).
- Suck with the mouth tap water at the TTS valve into the special pipette up to the uppermost ring mark (pressing the pinchcock at the same time) und place special pipette onto the distillation tube.
- Pipette 5.0 ml of the sample to be examined in den distillation flask.
- Add one spoon of pumice stones and one drop of antifoam solution.
- Connect distillation flask to the silicone plug of the distillation tube.
- Place volatile-acid measuring cylinder underneath the cooler.
- Ignite alcohol burner placing it in such a way underneath the distillation flask that the distance from the wick to the bottom of the flask is approx. 3 cm. Carry out distillation.
- Press the pinchcock of the pipette as soon as the distillate has reached the first ring mark of the volatile-acid measuring cylinders and allow water to drain until the next lower ring mark into the distillation flask; continue distillation until the second ring mark of the measuring cylinder, press the pinchcock of the pipette again and allow water to drain until the next lower ring mark of the pipette; continue distillation until the third ring mark of the measuring cylinder, drain the remaining water from the pipette into the distillation flask and continue distillation until the zero mark of the measuring cylinder. As soon as the zero mark has been reached, remove volatile-acid measuring cylinder and swing or extinguish alcohol burner.
- Add 1 drop of indicator solution to the distillate in the measuring cylinder.
- Add from the dropping bottle 1/100 n of caustic soda lye, while swivelling the measuring cylinder, until the emerging light red colouration of the liquid remains stable for approx. 30 seconds.
- Read the content of volatile acid in g/l at the volatile-acid measuring cylinder.

Important note:

1/100 n caustic soda lye is not stable enough for storage. It should be prepared in 2-weeks intervals from 1/10 n caustic soda lye. To do so, pipette 10.0 ml of 1/10 n caustic soda lye into a 100 ml volumetric flask, add distilled water exactly up to the ring mark and mix preparation thoroughly. Then fill the supply bottle for 1/100 n. Note production date on the supply bottle.

General remarks for the determination of volatile acid:

Contrary to the fruit acid contained in beverages, the volatile acid is, as its name indicates, volatile with water steam. For its determination, the beverage is therefore subjected to water steam distillation, and the acids gained in the distillate, mainly acetic acid apart from minor quantities of formic acid and propionic acid, are titrated by means of lye. In beverages with high SO_2 contents, the values achieved from the determination of the volatile acid are too high. For this reason, always determine the SO_2 contents in the respective beverage when achieving high values of volatile acid. The respective beverage with a high content of volatile acid should therefore also be subjected to a determination of the SO_2 content.

True volatile acid [g/l] = Titration value [g/l] – (0,001 x total SO_2 [mg/l])