

EN

ANNEX

**SPECIAL UNION ANALYSIS METHODS**

**ALLYL ISOTHIOCYANATE**

1. Principle of the method

Any allyl isothiocyanate present in the wine is collected by distillation and identified by gas chromatography.

2. Reagents

2.1. Ethanol, absolute.

2.2. Standard solution: solution of allyl isothiocyanate in absolute alcohol containing 15 mg of allyl isothiocyanate per litre.

2.3. Freezing mixture consisting of ethanol and dry ice (temperature – 60 °C).

3. Apparatus

3.1. Distillation apparatus as shown in the figure. A stream of nitrogen is passed continuously through the apparatus.

3.2. Heating mantle, thermostatically controlled.

3.3. Flowmeter.

3.4. Gas chromatograph fitted with a flame spectrophotometer detector equipped with a selective filter for sulphur compounds (wavelength = 394 nm) or any other suitable detector.

3.5. Stainless steel chromatograph column of internal diameter 3 mm and length 3 m filled with Carbowax 20M at 10 % on Chromosorb WHP, 80 to 100 mesh.

3.6. Microsyringe, 10µl.

4. Procedure

Put two litres of wine into the distillation flask, introduce a few millilitres of ethanol (point 2.1) into the two collecting tubes so that the porous parts of the gas dispersion rods are completely immersed. Cool the two tubes externally with the freezing mixture. Connect the flask to the collecting tubes and begin to flush the apparatus with nitrogen at a rate of three litres per hour. Heat the wine to 80 °C with the heating mantle, distil and collect 45 to 50 ml of the distillate.

Stabilize the chromatograph. It is recommended that the following conditions are used:

- injector temperature: 200 °C,
- column temperature: 130 °C,
- helium carrier gas flow rate: 20 ml per minute.

With the microsyringe, introduce a volume of the standard solution such that the peak corresponding to the allyl isothiocyanate can easily be identified on the gas chromatogram.

Similarly introduce an aliquot of the distillate into the chromatograph. Check that the retention time of the peak obtained corresponds with that of the peak of allyl isothiocyanate.

Under the conditions described above, compounds naturally present in the wine will not produce interfering peaks on the chromatogram of the sample solution.

Apparatus for distillation under a current of nitrogen

