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**Changes to legislation:** There are outstanding changes not yet made to Commission Regulation (EEC) No 2568/91. Any changes that have already been made to the legislation appear in the content and are referenced with annotations. (See end of Document for details) View outstanding changes

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## ANNEX XV

### 2. PROCEDURE

#### 2.1. Preparation of the test sample

If necessary, use the mechanical mill, which has previously been properly cleaned, to grind the laboratory sample in order to reduce it to particles that can pass completely through the sieve.

Use about one twentieth of the sample to complete the process of cleaning the mill, discard the ground material, grind the remainder and collect, mix carefully and analyze without delay.

#### 2.2. Test portion

As soon as the grinding operation has been completed, weigh out about 10 g of the sample to the nearest 0,01 g for testing.

#### 2.3. Preparation of the extraction thimble

Place the test portion in the thimble and plug with cotton wool. If a filter paper is used, envelope the test portion in it.

#### 2.4. Preliminary drying

If the olive residues are very moist (i.e., moisture and volatile matter content more than 10 %), carry out preliminary drying by placing the loaded thimble (or filter paper) in the oven heated for an appropriate time at not more than 80° C in order to reduce the moisture and volatile matter content to less than 10 %.

#### 2.5. Preparation of the round-bottomed flask

Weigh to the nearest 1 mg the flask containing one or two particles of pumice stone, previously dried in the stove at  $103 \pm 2^\circ$  C and then cooled in a dessicator for not less than one hour.

#### 2.6. Initial extraction

Into the extraction apparatus insert the thimble (or filter paper) containing the test portion. Pour into the flask the requisite quantity of hexane. Fit the flask to the extraction apparatus and place the whole on the electrically heated bath. Adjust the rate of heating in such a way that the reflux rate is not less than three drops per second (moderate, not violent boiling). After four hours extraction, allow to cool. Remove the thimble from the extraction apparatus and place it in a stream of air in order to drive off most of the impregnating solvent.

#### 2.7. Second extraction

Tip the contents of the thimble into the micro-grinder and grind as finely as possible. Return the ground mixture to the thimble without loss and place it back in the extraction apparatus.

Continue the extraction for a further two hours using the same round-bottomed flask containing the initial extract.

The resultant solution in the extraction flask must be clear. If not, filter it through a filter paper and wash the original flask and the filter paper several times with hexane. Collect the filtrate and the washing solvent in a second round-bottomed flask which has been dried and tared to the nearest 1 mg.

#### 2.8. Removal of solvent and weighing of extract

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Remove the greater part of the solvent by distillation on an electrically heated bath. Remove the last traces of solvent by heating the flask in the oven at  $103 \pm 2^\circ \text{C}$  for 20 minutes. Assist the elimination process either by blowing in air, or preferably an inert gas, at intervals or by using reduced pressure.

Leave the flask in a dessicator to cool for at least one hour and weigh to the nearest 1 mg.

Heat again for 10 minutes under the same conditions, cool in a dessicator and reweigh.

The difference between the two weighings shall not exceed 10 mg. If it does, heat again for periods of 10 minutes followed by cooling and weighing until the weight difference is 10 mg or less. Note the last weight of the flask.

Carry out duplicate determinations on the test sample.

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**Changes and effects yet to be applied to the whole legislation item and associated provisions**

- Signature words omitted by [S.I. 2019/1422 reg. 6\(10\)](#)
- Art. 1(8) inserted by [S.I. 2019/1422 reg. 6\(2\)\(b\)](#)
- Art. 1(8)(a)(ii)(bb) omitted in earlier amending provision S.I. 2019/1422, reg. 6(2)(b) by [S.I. 2020/1453 reg. 14\(16\)\(a\)\(i\)](#)
- Art. 1(8)(b) words substituted in earlier amending provision S.I. 2019/1422, reg. 6(2)(b) by [S.I. 2020/1453 reg. 14\(16\)\(a\)\(ii\)](#)
- Art. 1(8)(c)(ii) omitted in earlier amending provision S.I. 2019/1422, reg. 6(2)(b) by [S.I. 2020/1453 reg. 14\(16\)\(a\)\(iii\)](#)
- Annex 1a para. 1.1 words substituted by [S.I. 2019/1422 reg. 6\(11\)\(a\)](#)
- Annex 1a para. 1.2 words substituted by [S.I. 2019/1422 reg. 6\(11\)\(b\)](#)
- Art. 2a(3)(e) words substituted by [S.I. 2019/1422 reg. 6\(4\)\(c\)](#) (This amendment not applied to legislation.gov.uk. Reg. 6(4)(c) substituted immediately before IP completion day by S.I. 2020/1453, regs. 1(2)(b), 14(16)(c)(ii))
- Art. 2a(3)(e) words substituted by S.I. 2019/1422, reg. 6(4)(c) (as substituted) by [S.I. 2020/1453 reg. 14\(16\)\(c\)\(ii\)](#)
- Art. 3 words substituted by [S.I. 2019/1422 reg. 6\(5\)\(a\)](#)
- Art. 3 words substituted by [S.I. 2019/1422 reg. 6\(5\)\(b\)](#)