11.7.67 OFFICIAL JOURNAL OF THE EUROPEAN COMMUNITIES No 148/1

COUNCIL DIRECTIVE

of 27 June 1967

on the use of certain preservatives for the surface treatment of citrus fruit and on the control measures to be used for the qualitative and quantitative analysis of preservatives in and on citrus fruit

(67/427/EEC)

THE COUNCIL OF THE EUROPEAN ECONOMIC COMMUNITY,

Having regard to the Treaty establishing the European Economic Community, and in particular Article 100 thereof;

Having regard to the proposal from the Commission;

Having regard to the Opinion of the European Parliament[1];

Having regard to the Opinion of the Economic and Social Committee[2];

Whereas, under Article 5 of the Council Directive of 5 November 1963[3] on the approximation of the laws of the Member States concerning the preservatives authorised for use in foodstuffs intended for human consumption, as last amended by Article 1 of the Council Directive of 14 December 1966,[4] Member States may, until 30 June 1967, maintain in force the provisions of their national laws relating to the surface treatment of citrus fruit with biphenyl (diphenyl), orthophenylphenol and sodium orthophenylphenate;

Whereas the use of those substances for the surface treatment of citrus fruit does not constitute a danger to health where the residual amount per kilogramme of whole fruit does not exceed 70 milligrammes of biphenyl and 12 milligrammes of orthophenylphenol and sodium orthophenylphenate, expressed as orthophenylphenol;

Whereas, moreover, the treatment carried out should be indicated in an appropriate manner at all stages of marketing;

Whereas, if the three substances in question are to be authorised at Community level, common rules must be laid down for the official control of treated citrus fruit;

Whereas a transitional period is necessary before the provisions of this Directive can be implemented by the Member States; whereas the provisions of national laws relating to the surface treatment of citrus fruit with the three preservatives in question should therefore be maintained in force until the end of that period;

Whereas a Member State should not be required to authorise the use of a preservative in foodstuffs produced and consumed in its own territory when there is no technological reason to justify such use;

HAS ADOPTED THIS DIRECTIVE:

Article 1

The Council Directive of 5 November 1963 on the approximation of the laws of the Member States Concerning the preservatives authorised for use in foodstuffs for human consumption is hereby amended as follows:

1. In Article 2 (2), for the second sentence there shall be substituted the following:

> 'However, the laws of a Member State may totally exclude the use of any of the preservatives listed in the Annex only where there is no technological reason for using such preservative foodstuffs produced and consumed in its own territory.'

2. The following preservatives shall be added to those listed in Section I of the Annex:

[1] OJ No 63, 3.4.1967, p. 990/67.
[2] OJ No 64, 5.4.1967, p. 1005/67.
[3] OJ No 12, 27.1.1964, p. 161/64.
[4] OJ No 233, 20.12.1966, p. 3947/66

EEC No	Name	Conditions of use
E 230	Biphenyl (Diphenyl)	(a) Exclusively for surface treatment of citrus fruit; (b) At the time of marketing the citrus fruit (i) the residual amount per kg of citrus fruit (whole fruit) must not be greater than: for biphenyl: 70 mg, and for orthophenylphenol and sodium orthophenylphenate, used separately or together, expressed as orthophenylphenol: 12 mg;
E 231	Orthophenylphenol	(ii) the treatment must be indicated — in the wholesale trade, on invoices and on one external surface of the packaging, by the words: 'Preserved with...' giving the name of the substances used; — in the retail trade, by some visible indication giving the consumer clear information.
E 232	Sodium orthophenyl phenate	

3. Subparagraph (b) of Article 5 shall be deleted.

Article 2

The Member States shall take all measures necessary to ensure that the taking of samples and the qualitative and quantitative analysis of biphenyl, orthophenylphenol and sodium orthophenylphenate in and on citrus fruit are carried out in accordance with the provisions of Annexes I, II, III and IV to this Directive.

Article 3

1. Member States shall, not later than 1 July 1968, bring into force the measures necessary to comply with this Directive and shall forthwith inform the Commission thereof.

2. Until 1 July 1968 Member States may maintain in force the provisions of their national laws relating to the surface treatment of citrus fruit with biphenyl, orthophenylphenol and sodium orthophenylphenate.

Article 4

This Directive is addressed to the Member States.

Done at Brussels, 27 June 1967.

For the Council

The President

R. VAN ELSLANDE

ANNEX I

PROCEDURE FOR TAKING SAMPLES OF CITRUS FRUIT FOR PRESERVATIVE CONTROL

A. **Taking of samples**

I. Samples shall be taken using scientific methods which ensure that the samples are representative of the lot to be analysed.

II. The samples must satisfy at least the following requirements:

1. *Packaged goods* (crates, paperboard boxes, and similar containers)

Number of containers in the lot	Up to 20	From 21 to 500	From 501 to 1000	Above 1000
Minimum number of containers to be sampled	1	2	3	4
Mass, in kg, of fruit to be sampled per container	2	2	2	2

2. *Goods in bulk*

Mass of batch in kg	Up to 20	From 21 to 500	Above 500
Mass, in kg, to be sampled	2	4	8

III. By 'lot' is meant: a part of a consignment, which part has the same characteristics, such as variety, degree of ripeness, type of packaging.

B. **Packaging and delivery of samples**

1. The samples shall be placed in air-tight containers;

2. The containers shall be sealed;

3. The samples thus packaged shall be delivered as quickly as possible to the test laboratories.

ANNEX II

QUALITATIVE ANALYSIS FOR RESIDUES OF BIPHENYL, ORTHOPHENYLPHENOL AND SODIUM ORTHOPHENYLPHENATE IN CITRUS FRUIT PEEL

1. **Purpose and scope**

The method described below enables the presence of residues of biphenyl, orthophenylphenol (OPP) or sodium orthophenylphenate in the *peel* of citrus fruit to be detected. The sensitivity limit of this method, in absolute terms, is approximately 5 µg for biphenyl and 1 milligramme for OPP or sodium orthophenylphenate, which is the equivalent of 5 µg of biphenyl (5 ppm) and 1 milligramme of OPP (1 ppm) respectively in the peel of 1 kilogramme of citrus fruit.

When citrus fruit is treated with the above-mentioned products the residues deposited are found largely in the peel of the fruit. Quantitative analysis of such residues in the whole fruit therefore seems necessary only if they are found in the peel.

2. **Principle**

An extract is prepared from the peel using dichloromethane in an acid medium. The extract is concentrated and separated by thin layer chromatography using silica gel. The presence of biphenyl, orthophenylphenol or sodium orthophenylphenate is shown by fluorescence and colour tests.

3. **Reagents**

cyclohexane A.R.

dichloromethane A.R.

hydrochloric acid 25% (w/v)

silica gel GF 254 Merck or equivalent

0·5% solution of 2,4,7-trinitrofluorenone (Fluka, B.D.H or equivalent) (TNF) in acetone

0·1% solution of 2,6-dibromo-benzoquinone-4-chloroimide in ethanol (stable for up to one week if kept in the refrigerator)

concentrated solution of ammonia, S.G. : 0·9

standard 1% solution of pure biphenyl in cyclohexane

standard 1% solution of pure orthophenylphenol in cyclohexane

4. **Apparatus**

mixer

250 ml flask with ground glass joint and with a cooled reflux condenser

reduced pressure evaporator

micropipettes

thin layer chromatographic apparatus with plates measuring 20 × 20 cm

U.V. lamp (254 nm): the intensity should be such that a spot of 5 µg of biphenyl is visible

equipment for pulverising reagents

oven

5. **Method**

(a) *Preparation of the sample and extraction*

All the fruit in the sample for testing is cut in half. Half of each piece of fruit is kept for quantitative determination of residues of biphenyl and/or orthophenylphenol. Pieces of peel are taken from the other halves to give a sample of about 80 grammes. These pieces are chopped, crushed in the mixer and placed in the 250 ml flask; to this is added 1 ml of 25% hydrochloric acid and 100 ml of dichloromethane. The mixture is heated under reflux for ten minutes. After cooling and rinsing of the condenser with about 5 ml of dichloromethane, the mixture is filtered through a fluted filter. The solution is transferred to the evaporator and some porous granules are added. The solution is concentrated at reduced pressure at a temperature of 60 °C to a final volume of about 10 ml. If a rotary evaporator is used, the flask should be kept in a fixed position to avoid loss of biphenyl through the formation of a film of the product on the upper wall of the flask.

(b) *Chromatography*

30 grammes of silica gel and 60 ml of water are placed in a mixer and mixed for one minute. The mixture is then poured on to 5 chromatographic plates and spread to form a layer approximately 0·250 mm thick. The plates covered with this layer are subjected for fifteen minutes to a stream of hot air and then placed in an oven where they are kept for thirty minutes at a temperature of 110 °C.