Chlorides (2.4.4): maximum 500 ppm. Dilute 0.5 ml of solution S to 15 ml with water R. Prepare the standard using a mixture of 5 ml of chloride standard solution (5 ppm Cl) R and 10 ml of water R.

Sulphates (2.4.13): maximum 0.5 per cent. Dilute 0.3 ml of solution S to 15 ml with distilled water R.

Arsenic (2.4.2, method A): maximum 4 ppm, determined on 2.5 ml of solution S.

Heavy metals (2.4.8): maximum 40 ppm. Dilute 10 ml of solution S with dilute ammonia R1, using menthol yellow solution R as an external indicator. Dilute to 20 ml with water R and filter if necessary. 12 ml of this solution complies with test A. Prepare the reference solution using lead standard solution (2 ppm Pb) R.

Loss on ignition: 17 per cent to 34 per cent, determined on 0.5 g by ignition to constant mass at 900 ± 50 °C in a platinum crucible.

Acid-absorbing capacity. Suspend 0.25 g in 0.1 M hydrochloric acid, dilute to 100.0 ml with the same acid and allow to stand for 2 h in a water-bath at 37 ± 0.5 °C, with frequent shaking. Allow to cool. To 20.0 ml of the supernatant solution add 0.1 ml of bromophenol blue solution R and titrate with 0.1 M sodium hydroxide until a blue colour is obtained. The acid-absorbing capacity is not less than 100.0 ml of 0.1 M hydrochloric acid per gram.

ASSAY

Magnesium oxide. To 1.000 g in a 200 ml conical flask, add 35 ml of hydrochloric acid R and 60 ml of water R and heat in a water-bath for 15 min. Allow to cool, filter, wash the conical flask and the residue with water R and dilute the combined filtrate and washings to 250.0 ml with water R. Neutralise 50.0 ml of the solution with strong sodium hydroxide solution R (about 8 ml). Carry out the complexometric titration of magnesium (2.5.11). 1 ml of 0.1 M sodium edetate is equivalent to 4.030 mg of MgO.

Silicon dioxide. To 0.700 g add 10 ml of dilute sulphuric acid R and 10 ml of water R. Heat for 90 min on a water-bath with frequent shaking, replacing the evaporated water. Allow to cool and decant onto an ashless filter paper (diameter 7 cm). Wash the precipitate by decantation with 3 quantities, each of 5 ml, of hot water R, transfer it to the filter and wash it with hot water R until 1 ml of the filtrate remains clear after the addition of 0.05 ml of dilute hydrochloric acid R and 2 ml of barium chloride solution R1. Incinerate the filter and its contents in a platinum crucible, then ignite the residue (SiO₂) at 900 ± 50 °C to constant mass.

01/2008:1342

MAIZE OIL, REFINED

Maydis oleum raffinatum

DEFINITION

Refined maize oil is the fatty oil obtained from the seeds of Zea mays L. by expression or by extraction, then refined.

CHARACTERS

A clear, light yellow or yellow oil, practically insoluble in water and in alcohol, miscible with light petroleum (bp: 40 °C to 60 °C) and with methylene chloride.

It has a relative density of about 0.920 and a refractive index of about 1.474.

IDENTIFICATION

A. Carry out the identification of fatty oils by thin-layer chromatography (2.3.2). The chromatogram obtained with the test solution is similar to that obtained with the reference solution.

B. It complies with the test for composition of fatty acids (see Tests).

TESTS

Acid value (2.5.1). Not more than 0.5, determined on 10.0 g. If intended for use in the manufacture of parenteral dosage forms, not more than 0.3.

Peroxide value (2.5.5). Not more than 10.0. If intended for use in the manufacture of parenteral dosage forms, not more than 5.0.

Unsaponifiable matter (2.5.7). Not more than 2.8 per cent, determined on 5.0 g.

Alkaline impurities (2.4.19). It complies with the test for alkaline impurities in fatty oils.

Composition of fatty acids (2.4.22, Method A). The fatty-acid fraction of the oil has the following composition:

- fatty acids of chain length less than C₁₆: not more than 0.6 per cent,
- palmitic acid: 8.6 per cent to 16.5 per cent,
- stearic acid: not more than 3.3 per cent,
- oleic acid: 20.0 per cent to 42.2 per cent (equivalent chain length on polyethylene glycol adipate 18.3),
- linoleic acid: 39.4 per cent to 65.6 per cent (equivalent chain length on polyethylene glycol adipate 18.9),
- linolenic acid: 0.5 per cent to 1.5 per cent (equivalent chain length on polyethylene glycol adipate 19.7),
- arachidic acid: not more than 0.8 per cent,
- eicosanoic acid: not more than 0.5 per cent (equivalent chain length on polyethylene glycol adipate 20.3),
- behenic acid: not more than 0.5 per cent,
- other fatty acids: not more than 0.5 per cent.

Sterols. Determined by gas chromatography (2.4.23), the sterol fraction of the oil contains not more than 0.3 per cent of brassicasterol.

Water (2.5.32). If intended for use in the manufacture of parenteral dosage forms, not more than 0.1 per cent, determined on 5.00 g by the micro-determination of water. Use a mixture of equal volumes of decanol R and anhydrous methanol R as the solvent.

STORAGE

Store protected from light, at a temperature not exceeding 25 °C.

LABELLING

The label states:
- where applicable, that the substance is suitable for use in the manufacture of parenteral dosage forms,
- whether the oil is obtained by mechanical expression or by extraction.

01/2008:0344

MAIZE STARCH

Maydis amylum

DEFINITION

Maize starch is obtained from the caryopsis of Zea mays L.
CHARACTERS
Appearance: matt, white to slightly yellowish, very fine powder which creaks when pressed between the fingers.
Solubility: practically insoluble in cold water and in ethanol (96 per cent).
The presence of granules with cracks or irregularities on the edge is exceptional.

IDENTIFICATION
A. Examined under a microscope, using not less than 20 × magnification and using equal volumes of glycerol R and water R. It appears as either angular polyhedral granules of irregular sizes with diameters ranging from about 2 µm to about 23 µm or as rounded or spheroidal granules of irregular sizes with diameters ranging from about 25 µm to about 35 µm. The central hilum consists of a distinct cavity or two-to five-rayed cleft and there are no concentric striations. Between crossed nicol prisms, the starch granules show a distinct black cross intersecting at the hilum.
B. Suspend 1 g in 50 ml of water R, boil for 1 min and cool. A thin, cloudy mucilage is formed.
C. To 1 ml of the mucilage obtained in identification test B add 0.05 ml of iodine solution R1. An orange-red to dark blue colour is produced which disappears on heating.

TESTS
pH (2.2.3): 4.0 to 7.0.
Shake 5.0 g with 25.0 ml of carbon dioxide-free water R for 60 s. Allow to stand for 15 min.
Foreign matter. Examined under a microscope using a mixture of equal volumes of glycerol R and water R, not more than traces of matter other than starch granules are present. No starch grains of any other origin are present.
Oxidising substances (2.5.30): maximum 20 ppm, calculated as H2O2.
Sulphur dioxide (2.5.29): maximum 50 ppm.
Iron (2.4.9): maximum 10 ppm.
Shake 1.5 g with 15 ml of dilute hydrochloric acid R. Filter. The filtrate complies with the limit test for iron.
Loss on drying (2.2.32): maximum 15.0 per cent, determined on 1.000 g by drying in an oven at 130 °C for 90 min.
Sulphated ash (2.4.14): maximum 0.6 per cent, determined on 1 g.
Microbial contamination. Total viable aerobic count (2.6.12) not more than 10³ bacteria and 10² fungi per gram, determined by plate count. It complies with the test for Escherichia coli (2.6.13).

01/2008:1343

MALATHION
Malathionum

Diethyl (2RS)-2-(dimethoxyphosphinodithioyl)butanedioate.
Content: 98.0 per cent to 102.0 per cent (anhydrous substance).

CHARACTERS
Appearance: clear, colourless or slightly yellowish liquid.
Solubility: slightly soluble in water, miscible with acetone, with cyclohexane, with ethanol (96 per cent) and with vegetable oils.
It solidifies at about 3 °C.

IDENTIFICATION
Infrared absorption spectrophotometry (2.2.24).
Comparison: malathion CRS.

TESTS
Relative density (2.2.5): 1.220 to 1.240.
Optical rotation (2.2.7): −0.1° to + 0.1°.
Dissolve 2.50 g in ethanol (96 per cent) R and dilute to 25.0 ml with the same solvent.
Related substances. Liquid chromatography (2.2.29).
Solvent mixture: water R, acetonitrile R (1:3 V/V).
Test solution (a). Dissolve 0.10 g of the substance to be examined in the solvent mixture and dilute to 5.0 ml with the solvent mixture.
Test solution (b). Dilute 1.0 ml of test solution (a) to 10.0 ml with the solvent mixture.
Reference solution (a). Dissolve 0.100 g of malathion CRS in the solvent mixture and dilute to 50.0 ml with the solvent mixture.
Reference solution (b). Dilute 0.5 ml of test solution (a) to 100.0 ml with the solvent mixture.
Reference solution (c). Dissolve 5.0 mg of malathion impurity A CRS and 5.0 mg of malathion impurity B CRS in the solvent mixture, then dilute 50.0 ml with the solvent mixture.
Reference solution (d). Dilute 2.0 ml of reference solution (c) to 10.0 ml with the solvent mixture.

Column:
– size: l = 1.0 mm, Ø = 4.6 mm;
– stationary phase: octadecylsilica gel for chromatography R (10 µm);
– temperature: 35 °C.
Flow rate: 1 ml/min.
Detection: spectrophotometer at 210 nm.
Injection: 20 µl of test solution (a) and reference solutions (b), (c) and (d).
Retention time: impurity B = about 3.5 min; impurity A = about 5 min; malathion = about 16 min.
System suitability: reference solution (c):
– resolution: minimum 2.0 between the peaks due to impurities B and A.
Limits:
– impurity A: not more than 3 times the area of the corresponding peak in the chromatogram obtained with reference solution (d) (0.3 per cent);
– impurity B: not more than the area of the corresponding peak in the chromatogram obtained with reference solution (d) (0.1 per cent).