ETHYL METHYL KETONE

Prepared at the 28th JECFA (1984), published in FNP 31/2 (1984) and in FNP 52 (1992). No ADI was allocated at the 25th JECFA (1981)

SYNONYMS Methyl ethyl ketone, MEK

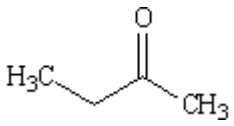
DEFINITION

Chemical names: 2-Butanone, butane-2-one

C.A.S. number: 78-93-3

Chemical formula: C_4H_8O

Structural formula:



Formula weight: 72.11

Assay: Not less than 99.5%

DESCRIPTION Clear colourless liquid with a characteristic odour

FUNCTIONAL USES Extraction solvent, flavouring agent (see "Flavouring agents" monograph JECFA no. 278)

CHARACTERISTICS

IDENTIFICATION

Solubility (Vol. 4): Freely soluble in water

Specific gravity (Vol. 4): d (20, 20): 0.803 - 0.807
d (25, 25): 0.801 - 0.803

PURITY

Distillation range (Vol. 4): 79 - 81°

Colour (Vol. 4): Not more than Colour Standard No. 10

Non-volatile residue (Vol. 4): Not more than 2 mg/100 ml

Water (Vol. 4): Not more than 0.2% (Karl Fischer Method)

Acidity: Not more than 0.003% (as acetic acid)
Transfer 75 ml (60 g) of sample into a 250-ml Erlenmeyer flask, add phenolphthalein TS, and titrate with 0.02 N alcoholic potassium hydroxide to a pink end-point that persists for at least 15 sec. Not more than 1.5 ml is required.

Hexan-2-one — Not more than 50 mg/kg
See description under TESTS

TESTS

PURITY TESTS

Hexan-2-one — Determine by *gas-liquid chromatography* (see Volume 4) using an instrument equipped with a flame ionization detector under conditions capable of an adequate separation of ethyl methyl ketone and hexan-2-one. Examples of appropriate operating conditions are:

(1) Column: 25 or 50 m oviod capillary column (methylpholysiloxane coating)
Column Temperature: 50^{o} - 100^{o} gradient with a temperature increase of 5^{o} per min.
(2) Column: 2 m x 2 mm O.D. glass column packed with 6.6 Carbowax 20 M on 80 - 120 mesh carbon black (supplied by Supelco, Inc.)
Column Temperature: 80^{o} - 180^{o} gradient with a temperature increase of 4^{o} per min.

In both cases nitrogen or helium carrier gas may be used at a flow rate of 20 - 30 ml/min and with an injection port temperature of 80^{o}.

Measure the areas under the peaks corresponding to ethyl methyl ketone and hexan-2-one and calculate the proportion of hexan-2-one.

METHOD OF ASSAY

Determine by *gas-liquid chromatography* (see Volume 4)(the GLC method allow the limit test for volatile organic impurities, such as alcohols, aldehydes, ketones; the total of these impurities shall not be more than 0.5%) using an instrument containing a thermal conductivity detector. Prepare a 4-m x 6-mm column consisting of a blend of equal quantities of 20% Carbowax 20 M on acid-washed, 60/80 mesh Chromosorb W, and 20% tetra-hydroxyethyl ethylenediamine on 30/60 mesh Chromosorb P, or use other suitable column materials capable of separating ethyl methyl ketone and the impurities whose retention times are listed below. Observe the following operating conditions during the determination:

Sample size: 10 µl
Column temperature: about 80^{o}
Helium flow rate: 30 to 32 ml per min
Detector voltage: 8.0 V
The approximate retention times, in min, are as follows:
acetone - 7
ethyl acetate - 9
ethyl methyl ketone - 11
tertiary-butanol - 14
methanol - 15
ethanol - 19
2-butanol - 30
propanol – 36

Measure the area under each peak of the chromatogram so obtained, calculate the area percent of each impurity, and record the sum of the impurities as A.

NOTE: In this procedure, area percent and weight percent may be assumed to be identical.
Calculate the percent of C_4H_8O in the sample by the formula:

$$100 - (A + B + C)$$

where
B = % of water determined under Water Content, and
C = % of acetic acid calculated from the test for acidity (in which each ml of 0.02 N alcoholic potassium hydroxide is equivalent to 1.20 mg of acetic acid).