6-Formyl-7,8-dihydropterin

Product number 11.416

Attention: 6-formylpterin is extremely (!) sensitive to light.

In a 1 l round bottom flask are filled 2.2 g of 6-formylpterin, 285 ml of EtOH anhydrous and 18.8 ml of HCl 37%. The flask is ultrasonicated and then 188 ml of triethylortho formate and a magnetic stirrer are added. The reaction mixture is mixed until all is dissolved and set aside overnight.
8.5 ml of piperidine are added (pH 8.5) and the flask is stored in a freezer for 3 hours. The reaction mixture is evaporated to dryness and afterward 15 ml of water are added. The residue is scraped up with a plastic rod and ultrasonicated.
The diethylacetal of 6-formylpterin is filtered, rinsed with water and dried in a vacuum desiccator over NaOH to give 2.8 g of diethylacetal of 6-formylpterin.

The equipment for hydrogenations is described in the chapter "General instructions for working with pteridines".

An egg shaped magnetic stirring rod, 2.0 g of diethyl acetal of 6-formylpterin, 825 ml of 0.01N NaOH and 750 mg of PtO_2 are filled in a 2 l three-necked round bottom flask of the hydrogenation equipment.
A dropping funnel with 16 ml of 1N hydrochloric acid is installed.
In order to remove all air, the equipment is twice evacuated and filled with nitrogen.
The equipment is evacuated, filled with hydrogen and then the mixture is stirred vigorously. After 3 hours the mixture fluoresces only weakly.
After 7 hours the 1N hydrochloric acid in the dropping funnel is added and the mixture is stirred for further 2 minutes.
The equipment is twice evacuated and filled with nitrogen.
The Pt is filtered out with a fine filter paper and rinsed with water.
The pH of the solution is about 3. The protective group is split off and the 6-formyl-tetrahydroperin is oxidized to 6-formyl-dihydropterin by the atmospheric oxygen.
The filtrate is poured in a crystallizing dish and slowly mixed with a magnetic stirring bar for 40 hours.
The crystallizing dish is covered and cooled to 0°C.
The precipitated 6-formyl-dihydropterin is filtered, rinsed with a small amount of water and dried in a vacuum desiccator over NaOH to give 600 mg of raw 6-formyl-dihydropterin.
The crude 6-formyl-7,8-dihydropterin is first purified by column chromatography and then by crystallization.
The column chromatography improves the color, the crystallization removes most impurities.

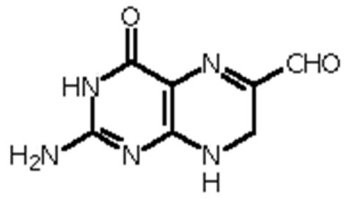
Preparation of the column:
Column diameter: 18 cm, height of the stationary phase: 22 cm
A slurry of water with cellulose MN 100 from Macherey-Nagel is poured through a funnel into the column. The top of the cellulose is protected by filter papers.

The column is washed with 0.2 N NaOH and then with water. The eluents must be pumped slowly through the column, otherwise the cellulose is compressed and the level of the cellulose sinks considerably.
The crude 6-formyl-7,8-dihydropterin (600 mg) is filled in a 6 l round bottom flask and 4.2 l of water are added. The flask is evacuated, stirred and warmed in a water bath at 60°C. The solution is cooled to about 40°C, filtered and slowly pumped on the top of the cellulose and subsequently the eluent water is pumped through the column.
The column and all 6-formyl-7,8-dihyropterin containing solutions must be protected from light.
The fractions are examined by TLC or HPLC.
The best fractions are collected, evaporated to about 1 l and cooled to 0°C overnight. The precipitated 6-formyl-dihydropterin is filtered, rinsed with water and dried in a vacuum desiccator over NaOH to give 245 mg of 6-formyl-dihydropterin.

Purity: 97.5% (HPLC)

Description: yellow powder

Solubility: 8 mg/100 g water (22°C)



6-Formyl-7,8-dihydropterin

$C_7H_7N_5O_2$
193.16
C 43.53% H 3.65% N 36.26% O 16.57%
Product no. 11.416

HPLC:

Sample	1 mg/5 ml 0.01 N NaOH
Column	Waters Spherisorb S5-ODS 1
Eluent	10 mM PB pH 6.0 plus10% MeCN
Flow	1 ml/min
Detection	254 nm

TLC:

TLC foil	cellulose 400 UV254
Developing solvent	3% Na2HPO4

The spots can be visualized by using ultraviolet light