Isoxanthopterin

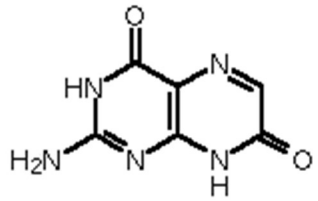
Product number 11.723 CAS number 529-69-1

In a 2 l round bottom flask with a magnetic stir bar are poured 7.15 g of ethyl glyoxylate (50% in toluene), 450 ml of water and 15 ml of toluene. Then a solution of 7.5 g of 2,4,5-triamino-6-hydroxypyrimidine dihydrochloride in 750 ml of water are added. A glass stopcock is installed and the round bottom flask is evacuated and the suspension is stirred vigorously for 1 hour.
Slowly a slightly yellow precipitation is formed.
A solution of 5.5 g of NH4OAc in 110 ml of water is added, the flask is evacuated and vigorously stirred.
After 3 hours the precursor of isoxanthopterin is filtered through a 17 cm filtering funnel. The filter cake is rinsed with 100 ml of water.

A 2 l round bottom flask with 2 l of water is put in a heating hood and the water is heated to boiling point. 28 ml of ammonia solution and then the wet filter cake of the precursor are added. The solution is boiled strongly for about one hour. The round bottom flask must be left open so that the ammonia can volatilize.
The solution is allowed to cool to 40°C.
The precipitated isoxanthopterin is filtered through a 7 cm filtering funnel. The filter cake is rinsed with 100 ml of water.

The wet isoxanthopterin is placed in a 2 l round bottom flask and a magnetic stir bar and 1 l of water are added. In order to dissolve the isoxanthopterin, 2 N NaOH is added (pH 11.8). 8 g of activated carbon are added and the mixture is stirred for 20 minutes.
The mixture is filtered through a 10 cm filtering funnel.
The filtrate is poured in a 2 l round bottom flask containing a magnetic stir bar and heated to boiling point. To the filtrate is added with stirring slowly a 3% solution of acetic acid until a pH of about 8 is reached.
The solution is allowed to cool to about 40°C.
The precipitated isoxanthopterin is filtered through a 10 cm filtering funnel. The filter cake is rinsed with 100 ml of water and then dried in a vacuum desiccator over NaOH to give 3.2 g of isoxanthopterin.

Purity:	99.4% (HPLC)
Description:	white to light beige powder
Solubility:	10 mg/100 ml of water (22°C)



Isoxanthopterin

$C_6H_5N_5O_2$
179.13

C 40.23% H 2.81% N 39.09% O 17.86%
Product no. 11.723

HPLC conditions:

Sample:	1 mg/5 ml 0.01 N NaOH
Column:	Spherisorb S5 ODS1, 150 mm x 4.6 mm
Eluant:	25 mM Na_2HPO_4, pH 7.5
Flow:	1 ml/min
Detection:	254 nm

The determination of ash showed up to 10%. We cannot explain these results!
Recrystallisation from water did not improve the result.
This compound is suitably for qualitative work only.
Heterocyclic compounds are very stable at high temperatures. I think that it is possible that isoxanthopterin does not completely disintegrate in the ash test at the usual temperatures.