Xanthopterin

Product number 11.713 CAS number 119-44-8

In a two-necked round bottom flask are poured 500 ml of H2O. The flask is cooled in an ice bath and 1000 ml of concentrated sulfuric acid are carefully *added*.
118 g of 2,4,5-triamino-6-hydroxypyrimidine sulfate are added and dissolved by mixing with a plastic rod. The three-necked round bottom flask is placed in an oil bath and the solution is slowly heated to about 80°C. 100 g of glyoxylic acid monohydrate are added and the solution is mixed and heated to about 100°C. The solution turns black.
After 15 minutes the flask is removed from the oil bath and cooled in a water bath. 750 ml of water are added and the flask is again cooled in the water bath.
Afterwards the flask is placed in a freezer (-20°C).
After one day the flask is swirled and after two other days the precipitated xanthopterin sulphate is filtered through a sintered disc filter funnel and rinsed with 120 ml of H2SO4/H2O 1:2 (-20°C).

1. **Recrystallisation**

In a two-neck round-bottom flask are poured 250 ml of H2O. The flask is cooled in an ice bath and 500 ml of concentrated sulfuric acid are carefully added.
The wet xanthopterin sulfate is added and dissolved by heating in an oil bath. Subsequently the flask is removed from the oil bath. When the temperature has reached 50°C, 380 ml of water are added. The flask is left overnight at room temperature and then placed in a fridge.
After two days the precipitated xanthopterin sulphate is filtered through a 7 cm sintered disc filter funnel and rinsed with 200 ml of H2SO4/H2O 1:2 (3°C)

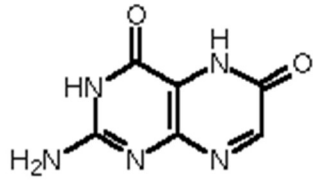
2. **Recrystallisation**

The wet xanthopterin sulphate is paced in a 10 l round bottom flask and 6 l of water are added. In order to dissolve the Xanthopterin 4 N NaOH is added (pH 11.8).
50 g of activated carbon are added and the mixture is stirred for 20 minutes.
The mixture is filtered through a 16 cm filtering funnel.
The filtrate is poured in a 10 l round bottom flask containing a magnetic stir bar. To the filtrate is added with stirring acetic acid (80%) until a pH of about 10 is reached.
The 10 l round bottom flask is put in a heating hood and the solution is heated to about 90°C and at the same time the pH is lowered by addition of diluted acetic acid with vigorous stirring to about 8.
The solution is allowed to cool overnight.
The pH is lowered by the addition of diluted acetic acid with vigorous stirring to 5.5 and left overnight.
The precipitated xanthopterin is filtered through a 17 cm filtering funnel. The filter cake is rinsed with 1.2 l of water and then dried in a vacuum desiccator over NaOH to give 28 g of xanthopterin.

Purity: 99.4%

Description: Yellow powder

The determination of ash showed up to 10%. We cannot explain this result!
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 xanthopterin does not completely disintegrate in the ash test at the usual temperatures.



Xanthopterin

$C_6H_5N_5O_2$
179.13

C 40.23% H 2.81% N 39.09% O 17.86%
Product no. 11.713

HPLC:

Column	Zirchrom PBB
Eluant	22 mM Na2HPO4 pH 7.5 - Methanol (4:1)
Flow (ml/min)	1
Wavelength (nm)	254
Conc.	0.5 mg/ml plus minimal ammonium hydroxide
Purity	>99.0%