VALIDATED, STABILITY-INDICATED QUANTITATIVE PURITY TESTFOR TRIETHYLENETETRAMINE TETRACHLORHYDRATE BY AMD.

D.POIRIER[2], C.DAUPHIN[1], D.PRADEAU[1].
1-AGEPS,Laboratoire de développement analytique,7 rue du fer à moulin,75221,Paris Cedex 5.
2-Travail effectué dans ce même laboratoire.

Capsules of triethylenetetramine tetrachlorhydrate (TETA,4HCl) are part of the "orphan" drug arsenal.This drug is used as second intention in Wilson disease that is characterized by defective copper elimination.

To guarantee the quality and safety of a starting material intended to be used as a medicine preparation, the impurities described in United States Pharmacopoeia are detected by automated multiple development.
Impurities analysis is made by 18-step isocratic development with the phase mobile: methanol/eau ultrapure/acétonitrile (5/1/4), the chamber is satured with 25% ammonia, the visualization is by ninhydrin and quantification by densitometry.

The impurities are : diethylenetriamine (impurity B)
1-(2-aminoethyl)piperazine (impurity C)
tris(2-aminoethyl)amine (impurity D)

The identification of these impurities by USP requires two different mobile phases.

Statistic parameters determined during validation are entirely acceptable for the detection and quantification by densitometry of the three impurities.
Linearity at J1, J2 and J3 was good for impurities C and D and less for impurity B because of greater diffusion giving a higher limit of detection (about 27ng) while those of impurities C and D are 7.5 and 3.5ng.
Specificity is good, reliability and accuracy are acceptable.