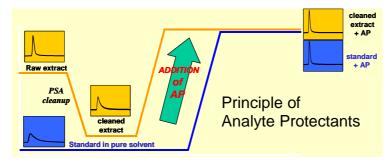


Single Residue Methods

What to pay attention to when using Analyte Protectants (AP)

AP only work for GC analysis: Analyte Protectants (AP) are only designed for GC analysis, not for LC (do not function in LC analysis). APs with hydroxyl groups, e.g. sugars and sugar derivates, have shown to be especially effective, since they can block active sites in the GC system via hydrogen bonds. When using APs, notably narrower peaks can be obtained for many substances. Thus, the chromatographic separation is improved, lower detection limits are achieved and the determination of the peak area is simplified and more precise.



Preparation of the AP mix:

The concentrations of the AP mix in acetonitrile:water solvent mixture are:

sorbitol 5 mg/mL (protects late eluting substances),

3-ethoxy-1,2-propanediol 200 mg/mL (protects early eluting substances),

D-(+)-gluconic acid-δ-lactone 10 mg/mL (protects early and middle fast eluting substances), shikimic acid 5 mg/mL (protects base-labile substances such as captan, folpet, tolylfluanid, and dichlofluanid).

Usually 30 µL of AP mix are employed per 1 mL final sample extract or solvent.

Preparation of stock solutions:

- Sorbitol (CAS Nr. 50-70-4): dissolve 500 mg in 10 ml 10 ml acetonitrile:water (1:1 v:v)
- D-(+)-gluconic acid-δ-lactone (CAS Nr. 90-80-2): dissolve 500 mg in 10 ml acetonitrile:water (6:4 v:v)
- Shikimic acid (CAS Nr. 138-59-0): dissolve 500 mg in 10 ml acetonitrile:water (6:4 v:v)
- 3-Ethoxy-1,2-propanediol (CAS Nr: 1874-62-0): use undissolved

Preparation of AP mix:

- Weigh 2 g of 3-ethoxy-1,2-propanediol in a 10 ml volumetric flask.
- Add 2 ml D-(+)-gluconic acid-δ-lactone stock solution, 1 mL sorbitol stock solution and 1 mL shikimic acid stock solution.
- Fill the flask with acetonitrile:water (7:3 v:v) to the 10 mL mark (store at 4° C).

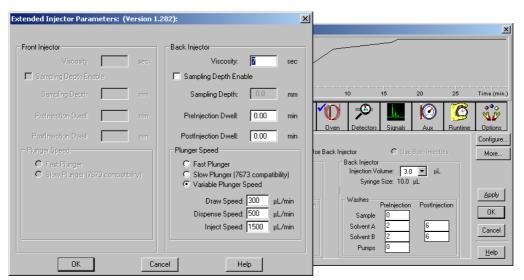


Single Residue Methods

Rinsing of the GC syringe: when using AP, the syringe of the autosampler might get stuck - thus, it is important to rinse the syringe thoroughly with water or a water-containing polar solvent mix after each injection (see exemplary injection parameters). Depending on the autosampler, manual cleansing of the syringe stamp after about 100 injections might be additionally necessary. AP-remains dissolve best in water or other polar solvents.

Example 1: injection parameters for Agilent 6890 MS 5973N with auto sampler

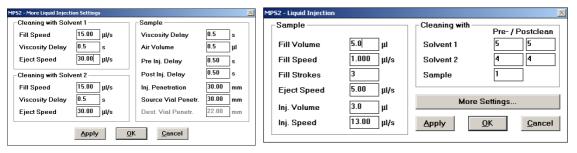
Solvent A: water Solvent B: isobutanol



Example 2: injection parameters for Waters Quattro micro with CTC Combi PAL Autosampler Gerstel-MPS2:

Solvent 1: MeOH / H₂O (80:20)

Solvent 2: acetone



Check for interferences: in GC-MS analysis via SIM-mode interferences play a minor role, since acquisition of selected masses is performed. But when using less specific detectors, e.g. ECD or NPD, interferences may occur more often - thus check for them. Also check for interferences when using GC-TOF or MS-fullscans, since high concentrations of AP may blanket the identification of pesticides.