

## Determination of hexachlorocyclohexane isomers and chloro substituted benzene congeners in air

Syncore® Analyst R-12, Residual volume 1.0 mL, Vacuum Pump V-300, Interface I-300 Pro, Recirculating Chiller F-314, Flushback module R-12: Time saving concentration and reproducible recovery rates of volatile analytes.

In the past, different hexachlorocyclohexane isomers and chloro substituted benzenes were produced and industrially applied in large quantities. Many of these substances have been deposited, accumulated and are still released in the environment from various sources today. The contaminants can be routed back to incomplete combustion, leaching of landfill sites or inadequate production of chlorinated compounds or pesticides. [1] Analytical contract laboratories, usually concentrate environmental samples from many different matrices prior to the analysis by GC-MS. *mas I münster analytical solutions gmbh*, established methods for the determination of these contaminants from environmental screenings.

In this short note, the use of the Syncore® as a time saving and reliable instrument for the determination of HCH and chloro-substituted benzenes in ambient air is illustrated.

### 1. Introduction

The quantitative determination of these halogenated analytes requires sophisticated GC-MS measurements. For the quantification, internal standards are used to overcome sample-volume variations, different effects of sample preparation and instrument drift. Internal standards consist of similar molecules in known concentrations that are added to the sample and allow quantification of the undetermined substance. The signal of the analyte is then compared with the signal of added standard to quantify the analyte.

The following equation can be used for the quantification of the unknown analytes:

$$\frac{A_x}{C_x} = \frac{A_{IS}}{C_{IS}} \cdot F$$

- $A_x$  peak area sample
- $A_{IS}$  peak area internal standard
- $C_x$  concentration sample
- $C_{IS}$  concentration internal standard
- $F$  response factor

In a first step, the equation is solved for the response factor  $F$  with a known analyte concentration and the internal standard. The same equation can be used to calculate an unknown sample concentration with the previously determined response factor  $F$ .

In the following paragraphs, the use of a Syncore® Analyst for the parallel solvent concentration to a residual volume is shown in detail.

### 2. Experimental

The air sample collection is performed over a 24 h period on TENAX® absorption tubes. The corresponding <sup>13</sup>C labeled radio-isomers are added as internal standards. The extraction of the content is then performed 3 times with 8 mL of n-hexane using an ultrasonic bath (60 °C, 15 min). Each extract is directly filtered into the Analyst vessel through a 0.45 µm PTFE filter. 1 mL of toluene is added as a keeper solvent and the volume reduced by means of following equipment:

Syncore® Analyst R-12, residual volume 1.0 mL with primary S-condenser / Vacuum Pump V-300 / Interface I-300 Pro / Recirculating Chiller F-314.

The residual volume is transferred into standard GC vial after evaporation. The quantification of the analytes was carried out by GC-MS/MS (triple quadrupole).

Table 1: Syncore® Analyst R-12 settings

Platform temperature	45 °C
Coolant temperature	10 °C
Vacuum cover temperature	45 °C
Rotation speed	220 rpm
Time	15 min
Pressure	Isocratic: 150 mbar

Table 1 shows the settings for the evaporation step using the Syncore® Analyst R-12. This set-up allows a semi-automated and fast concentration of the 24 mL n-hexane extract to a residual volume of approx. 1.0 mL.

### 3. Results

The recovery of the highly volatile analytes is consistently above 90% and shows high reproducibility. The concentration to the predefined volume is achieved based on to the chilled appendix technology. The accuracy of the residual volume does not show to be a crucial factor as internal standards are commonly added and allow quantitative evaluation. Analyte recoveries are increased by the use of a Flushback module that optimizes the concentration process. Furthermore, the evaporated solvents can be recollected and recycled at a rate of up to 95%.

### 4. Conclusion

The determination of organic contaminants normally requires multiple sample preparation steps. In most cases, large amounts of solvents have to be concentrated. The processing of large sample series show, that parallel concentration of extracts offers a significant reduction of costs and increased handling efficiency. Since internal standards were used, the residual volume after evaporation was found to be a none critical factor.

Due to the high volatility of the analytes the use of a parallel nitrogen blow down evaporators (Kurderna-Danish) was found to be unfeasible due to the high potential of cross contamination and analyte loss.

Thus, the Syncore® Analyst is a versatile and economic solution for the effective parallel solvent evaporation of extracted environmental samples.

### 5. Acknowledgement

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### 6. References

- [1] Fiedler H.(1993), *Stoffbericht Hexachlorcyclohexan (HCH)*, Texte und Berichte zur Altlastenbearbeitung