

THE EFFECT OF EVAPORATION CONDITIONS AND KEEPER SOLVENTS ON LOSSES OF TETRACHLORODIBENZODIOXIN DURING CONCENTRATION

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INTRODUCTION

Most schemes of analysis for PCDDs and PCDFs require evaporation of extracts to very small volumes or to dryness, either for solvent exchange prior to the next clean-up step or for final concentration prior to GC-MS determination. In our laboratory we carry out concentration by "blow-down" using a gentle jet of nitrogen gas directed at the surface of the extract contained in a small tube or vial.

There is a potential risk of solute loss during such steps. Several reports note that care is needed,^{1,2,3} and O'Keefe has investigated losses using a specialised apparatus.⁴ A number of workers recommend the use of a small quantity of a low volatility "keeper" solvent to reduce losses.^{5,6} In the past our practice has been to add tetradecane (5 μ l) prior to evaporation. Unfortunately when the residue of keeper solvent is diluted with a more volatile solvent for GC/MS analysis, the mixed solvent system can have an adverse effect on the chromatography leading to peak broadening.

To check whether such keeper solvents are really necessary and to achieve optimum conditions for blow-down we have carried out a series of experiments using ¹⁴C-labelled 2,3,7,8-TCDD and monitoring recoveries by liquid scintillation counting.

EXPERIMENTAL

Samples of ¹⁴C-TCDD (2.5ng) were prepared in 1.00mL of dichloromethane, cyclohexane or toluene. Solutions were blown down in 1.1ml tapered glass vials (type 1.1-STVG; Chromacol Ltd, UK) using a Pierce Reactitherm Apparatus consisting of a thermostatted alloy heating block, and an overhead gas delivery manifold fitted with 10swg stainless steel needles. Nitrogen was applied at 0.15-0.2 bar pressure. In one series of experiments vials were placed in wells drilled to accommodate the entire vial with a fairly close fit. In other experiments vials were supported above an undrilled block with a Perspex guide so that only a small area of glass was in contact with the block.

Following evaporation samples were reconstituted in either (a) 1.00mL solvent followed by 2 x 0.50mL rinses to ensure complete recovery of material dispersed over the vial surface, or (b) 25 μ l n-nonane only to represent normal practice immediately prior to GC/MS.

The reconstituted samples were transferred to scintillation vials. For the samples transferred in 25 μ l n-nonane, 2.00mL of the appropriate solvent was added to the vial to allow for solvent effects on scintillation. Scintillant (Fluoran hv, 10.0mL) was added and the samples were counted (LKB Rackbeta

1216). Recoveries were calculated by comparison of sample counts per minute (CPM) with the mean CPM of a control group which was not blown down.

RESULTS

Table 1. Recovery (%) of TCDD following blow-down with vial in well of heater block and addition of 1ml solvent.

| Procedure | Temperature of block | 30°C | 50°C | 70°C | 90°C |
|--------------------------------|----------------------|-------|-------|-------|------|
| dichloromethane with keeper | | 102.3 | 102.1 | 99.1 | - |
| dichloromethane without keeper | | 102.2 | 104.4 | 101.5 | - |
| cyclohexane with keeper | | 94.7 | 96.8 | 95.7 | - |
| cyclohexane without keeper | | 97.5 | 95.2 | 94.9 | - |
| toluene with keeper | | - | 99.6 | 99.6 | 97.5 |
| toluene without keeper | | - | 96.6 | 68.8 | 46.2 |

Table 2. Recovery (%) of TCDD following blow-down with vial above heater block and addition of 1ml solvent.

| Procedure | Temperature of block | 30°C | 50°C | 70°C | 90°C |
|--------------------------------|----------------------|------|------|------|------|
| dichloromethane with keeper | | 96.7 | 95.3 | 96.1 | - |
| dichloromethane without keeper | | 97.6 | 96.7 | 97.1 | - |
| cyclohexane with keeper | | 97.4 | 98.3 | 99.2 | - |
| cyclohexane without keeper | | 99.5 | 96.9 | 98.2 | - |
| toluene with keeper | | - | 97.9 | 99.2 | 99.2 |
| toluene without keeper | | - | 97.9 | 96.1 | 88.7 |

Table 3. Recovery (%) of TCDD following blow-down with vial in well of heater block and addition of 25ul solvent.

| Procedure | Temperature of block | 30°C | 50°C | 70°C | 90°C |
|--------------------------------|----------------------|------|------|------|------|
| dichloromethane with keeper | | 93.4 | 89.5 | 92.6 | - |
| dichloromethane without keeper | | 88.5 | 86.3 | 63.8 | - |
| cyclohexane with keeper | | 90.5 | 87.0 | 81.8 | - |
| cyclohexane without keeper | | 91.2 | 89.1 | 70.0 | - |
| toluene with keeper | | - | 92.1 | 88.7 | 89.3 |
| toluene without keeper | | - | 90.1 | 84.2 | 79.1 |

DISCUSSION

The results in table 1 show that good recoveries can be obtained without keeper solvent, even when much of the surface of the vial is in contact with the heater block. There is, however, clear evidence of

losses when toluene is evaporated with block temperatures of 70°C or 90°C. In these experiments the vials were carefully monitored and removed as soon as the solvent was fully evaporated. However it is probable that the minor inconsistencies in the data presented in tables 1 and 3 regarding the onset and extent of losses are related to variations in the length of time that heat and gas flow were maintained after full evaporation. The losses under the more extreme conditions are reduced when keeper solvent is used. The results in table 2 show that even these losses can be reduced by placing only the tip of the vial in contact with the heater block. Observation of the liquid level is also much easier with this arrangement. This is, however, less important since we find that blow-down can be continued for a reasonable period after complete removal of the solvent without loss of TCDD.

The results in table 3, where only 25ul of solvent was used, should be compared with those in table 1. The generally slightly lower and more variable recoveries presumably reflect the difficulty of ensuring that the small solvent volume wets all of the inner surface of the vial over which the solute may be distributed.

CONCLUSIONS

Dilute solutions of TCDD can safely be reduced to dryness by blow-down without using keeper solvents as long as the temperature is not excessive. Resting the tip of a conical vial on a hot plate at temperatures up to 70°C provides sufficient heat to prevent slowing of evaporation or cooling and condensation of moisture, but avoids risk of solute loss.

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