

**USING A VACUUM CENTRIFUGE TO REPLACE
NITROGEN BLOW DOWN IN SAMPLE PREPARATION**

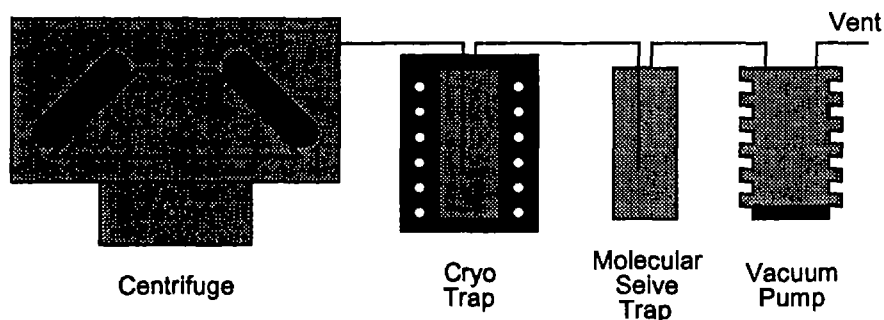
Bruce N. Colby & C. Steve Parsons
Pacific Analytical
6349 Paseo Del Lago
Carlsbad, California 92009 USA

Nearly all methods for the determination of chlorinated dioxins and furans involve some form of solvent extraction. The resulting extracts are reduced in volume by about a factor of up to 400,000 prior to analysis. Solvent volumes are normally reduced using some combination of Kuderna-Danish (K-D) distillation, rotary evaporation and nitrogen blow down. Each of these techniques has advantages and disadvantages which depend on initial volume, desired final volume and the solvent's boiling point. K-D for example, is very effective with large initial volumes of low boiling solvents such as methylene chloride (bp 40°C) but it is impractical when used with higher boiling solvents such as toluene (bp 111°C) or when a small final volume is required. Rotary evaporation is also effective with large initial solvent volumes plus it works well with higher boiling solvents such as toluene. However, it is not practical when small final volumes are needed due to the large surface area of the glassware involved. Nitrogen blow down is not practical with large or even medium volumes of high boiling solvents due to the time required but it is the only effective technique when small final volumes are required.

When environmental samples are prepared for dioxins and furans analysis in our laboratory, the last solvent reduction steps has involved taking 20 mL of toluene to dryness via nitrogen blow down before taking the residue up in 1 mL of toluene and reducing to a final volume of 10 µL, again using nitrogen blow down. Reducing 20 mL to dryness using nitrogen blow down takes about two and one half hours or approximately 60 percent of the total time spent reducing solvent volumes. In an attempt to reduce the time associated with this step, we recently undertook a study where we replaced the nitrogen blow down with vacuum centrifugation. It resulted in a 40 percent reduction in the total time spent reducing solvent volumes plus it had several additional benefits. First, the skill level required of the analyst was reduced because there was no chance for cross contamination resulting from splashing between vials in a heated multi-sample block and because there was no gas stream which could blow crystallized material out of the sample tube. Second, the waste toluene was cryogenically trapped rather than vented into the laboratory atmosphere or up a hood to cause air pollution.

The vacuum centrifuge tested was a Labconco Centrivap (Figure 1). It utilizes a 5 position sample head which rotates at 1800 rpm. Each position holds a 50 mL sample tube. It is evacuated using an Edwards 5 mechanical pump which generates a vacuum of 750 mm Hg. All solvent vapors removed from the extracts are trapped using the integral cryogenic cooling unit. In the experiments described here, 20 mL aliquots of toluene were reduced in the Centrivap as follows. After placing the sample vials in the centrifuge head, the vacuum pump and rotor were turned on. After 15 minutes, the device was stopped and the solvent level was checked. If solvent remained, another 15 minute cycle was initiated. This process was repeated until the samples had been taken to dryness. The total time required was 55 minutes. After solvent reduction, 1 mL of toluene was added to each sample tube, then the tube was capped and vortexed for 30 sec. The solution was then transferred to a conical vial, internal standard was added and the volume was reduced to 10 µL prior to analysis by HRGC/HRMS (VG 70VSE).

Figure 1 - Vacuum Centrifuge



Two experiments were performed. First, five 10 mL aliquots of toluene were spiked with 2,3,7,8-TCDD and 2,3,7,8-TCDF at 0.1 ng/mL. These were concentrated and analyzed as described above and percent recoveries were calculated. The results shown in Table 1 clearly indicate that good recovery and precision are achieved with the Centrivap. Also, because no dioxins or furans were detected in the blank, cross contamination was not a problem.

Table 1 - Chlorinated Dioxin and Furan Recoveries

Analyte	R1	R2	R3	R4	R5	Mean %sd
2,3,7,8-TCDD	100	102	117	96	102	103 7.7
2,3,7,8-TCDF	100	113	120	106	101	108 7.8

In the second experiment, a set of EPA Method 8290 calibration standards was subjected to 100 fold dilution in toluene followed by concentration back to original volume in the Centrivap. The purpose of this experiment was to establish that no concentration dependent problems would be encountered. As can be seen from the resulting relative response factors in Table 2, there is no concentration related trend associated with using the Centrivap.

Finally, it is worth noting that after six months of continuous operation with dioxin field and QC sample preparations, no cross contamination or blank levels have been noted. Clearly, the vacuum centrifuge is an easy, effective and efficient means for removing toluene from sample extracts. It is also significant that air pollution from waste solvent is minimized.

Table 2 - Calibration Standards Results

Analyte	RRF					Mean	%sd
	CC1	CC2	CC3	CC4	CC5		
2378-TCDD	1.400	1.150	1.270	1.308	1.388	1.303	7.8
2378-TCDF	0.800	0.950	1.120	1.185	1.134	1.038	15.4
12378-PeCDF	0.880	0.970	1.028	1.051	1.171	1.020	10.5
12378-PeCDD	1.120	1.100	1.252	1.245	1.313	1.206	7.6
23478-PeCDF	0.840	0.790	1.094	1.049	1.007	0.956	14.0
123478-HxCDF	1.320	1.270	1.672	1.567	1.398	1.445	11.7
123678-HxCDF	1.240	1.170	1.622	1.526	1.293	1.370	14.2
123478-HxCDD	1.200	1.200	1.660	1.398	1.388	1.369	13.8
123678-HxCDD	0.920	0.910	1.098	1.102	1.017	1.009	9.2
123789-HxCDD	1.320	1.300	1.558	1.617	1.545	1.468	10.0
234678-HxCDF	1.200	1.240	1.452	1.512	1.336	1.348	9.9
123789-HxCDF	1.360	1.260	1.550	1.526	1.392	1.418	8.5
1234678-HpCDF	1.520	1.470	1.592	1.656	1.642	1.576	5.1
1234678-HpCDD	1.240	1.120	1.316	1.256	1.219	1.230	5.8
1234789-HpCDF	1.560	1.480	1.992	1.699	1.670	1.680	11.6
OCDD	1.600	1.240	1.490	1.326	1.294	1.390	10.8
OCDF	1.480	1.420	1.696	1.664	1.723	1.597	8.6

