

Full Automatic Clean-up Robot for Dioxin/PCB Analysis (II)

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Introduction

Dioxin analysis requires several steps of clean-up procedures by combination of several column chromatography (e.g. silica gel column chromatography, carbon column chromatography) and sulfuric acid treatment. Full Automatic Clean-up Robot for Dioxins and PCBs were developed (1).

Hardware

Robot is constituted by two apparatus. One is "Sulfuric Acid Treatment Unit" and another one is "Column Chromatography Clean-up Unit". Two apparatus can operate alone and/or combination.

Robot is constituted by syringe, pump, concentration flask with heater, stirrer, atomizer needle, needle cleaning port, column chromatograph, et al. These parts are assembled in X-Y dimensional arm (full view and several piece of robot are shown in **Photo-1 and Photo 2-5**).

To protect from line contamination, contentious on-line flow method was not employed in this system.

This robot treats six samples in parallel.

Accuracy and Precision

An example of reproducibility, results of repeatedly testing for sulfuric acid spiking is shown in **Table 1**. About 10mL conc. sulfuric acid were spike into glass flask, standard deviation were calculated for each line. Results represents SD (%) were less than 0.3%.

Table 2 represents the reproducibility for actual sample regarding three analytical stages, i.e. sulfuric acid treatment, multilayer silica gel column chromatography and active carbon silica gel column chromatography. SD (%) were less than 10% for all PCDDs/PCDFs isomers that have TEF (WHO-1998).

Table 1. An example of reproducibility for spike of sulfuric acid. (unit: g)

No. of Measurement	Line No.1	Line No.2	Line No.3	Line No.4	Line No.5	Line No.6
1	18.22	18.20	18.23	18.28	18.24	18.15
2	18.32	18.26	18.20	18.24	18.27	18.31
3	18.32	18.19	18.36	18.31	18.25	18.19
4	18.31	18.31	18.20	18.28	18.28	18.23
5	18.31	18.26	18.26	18.25	18.23	18.27
6	18.27	18.24	18.31	18.23	18.27	18.30
7	18.27	18.24	18.32	18.19	18.23	18.23
8	18.25	18.23	18.27	18.22	18.26	18.20
9	18.27	18.25	18.26	18.19	18.31	18.24
10	18.26	18.27	18.28	18.24	18.26	18.26
average	18.280	18.245	18.269	18.243	18.260	18.238
SD	0.0337	0.0344	0.0515	0.0389	0.0245	0.0496
SD (%)	0.184	0.189	0.282	0.213	0.134	0.272

Calculated Volume (mL) *	9.96	9.94	9.95	9.94	9.95	9.93
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* calculated from specific gravity of sulfuric acid (1.836)

Table 2. An example of reproducibility for three stages.

Compounds	Reproducibility (% of standard deviation)		
	Sulfuric acid treatment	Multilayer silica gel	AC silica gel
PCDDs 2,3,7,8-TeCDD	3.6	5.7	7.5
1,2,3,7,8-PeCDD	2.1	7.6	7.1
1,2,3,4,7,8- HxCDD	6.8	8.6	8.4
1,2,3,6,7,8- HxCDD	4.7	3.6	8.2
1,2,3,7,8,9- HxCDD	4.1	4.6	6.4
1,2,3,4,6,7,8- HpCDD	5.1	4.9	7.4
OCDD	6.2	10.0	8.3
PCDFs 2,3,7,8-TeCDF	2.1	7.6	7.8
1,2,3,7,8-PeCDF	4.0	6.2	6.6
2,3,4,7,8-PeCDF	1.9	5.1	7.2
1,2,3,4,7,8-HxCDF	4.5	4.6	7.6
1,2,3,6,7,8-HxCDF	5.4	6.3	10.0
1,2,3,7,8,9-HxCDF	4.1	5.2	9.1
2,3,4,6,7,8-HxCDF	5.2	6.3	9.0
1,2,3,4,6,7,8- HpCDF	6.6	4.3	8.9
1,2,3,4,7,8,9- HpCDF	4.1	6.6	8.3
OCDF	3.4	7.0	8.8

air filter

organic solvent

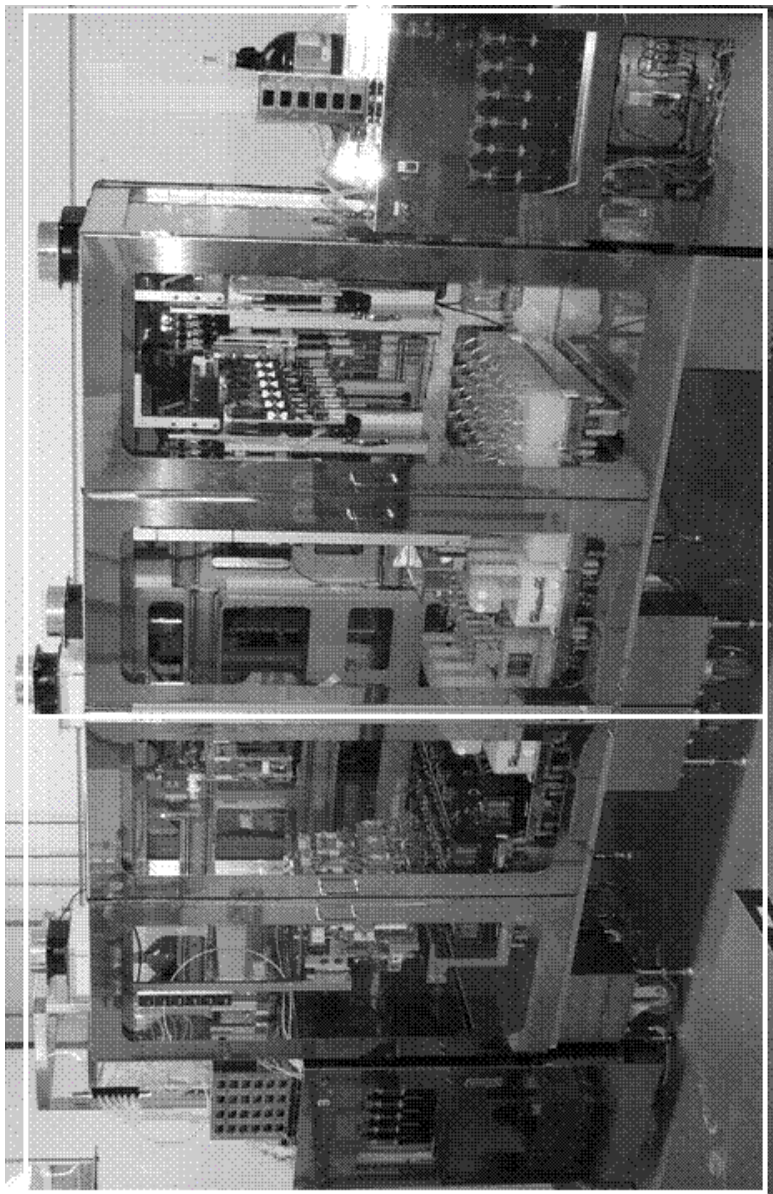


Photo 1. Full View of "Full Automatic Clean-up Robot for Dioxins and PCB's"

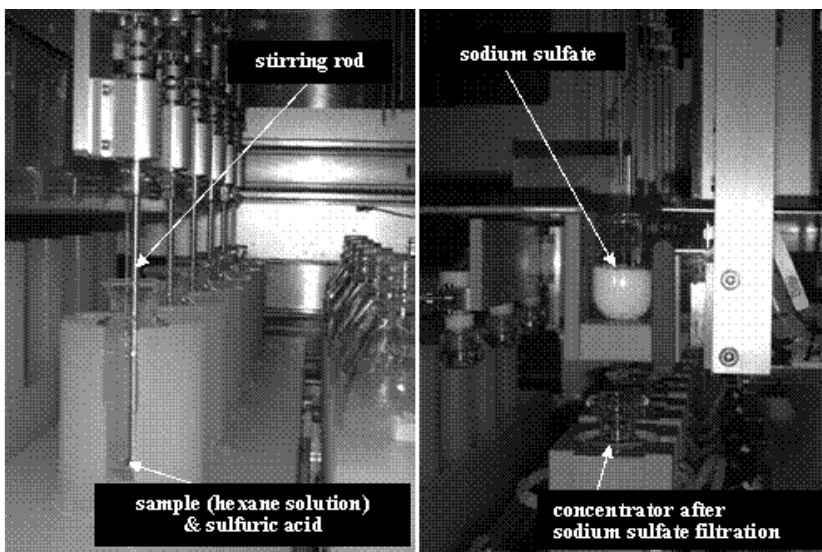


Photo 2. Stirring for sulfuric acid treatment.

Photo 3. Filtration after sulfuric acid treatment for dehydration (sodium

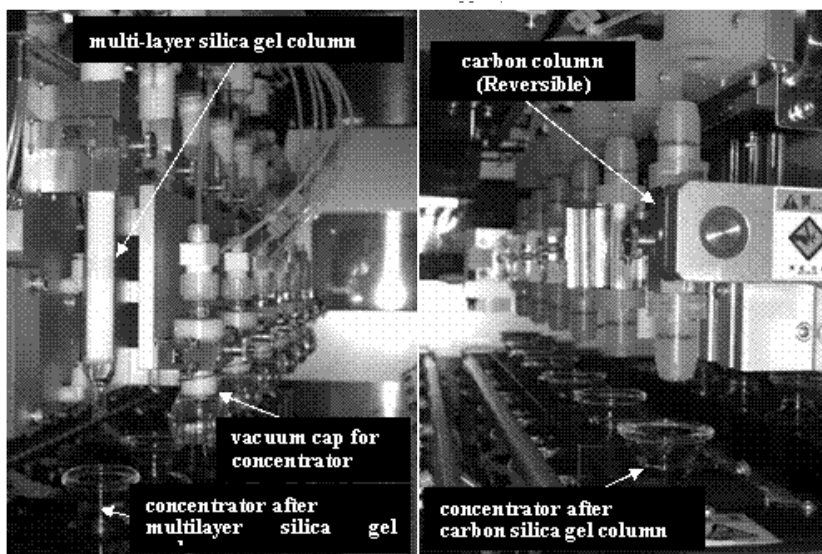


Photo 4. Multilayer silica gel column.

Photo 5. Carbon silica gel column .

Reference

- (1) Matsumura, T., Masuzaki, Y., Takahashi, A., Koizumi, A., Okuyama, H., Kawada, Y. and Higashiguchi, T. (2004) Full Automatic Clean-up Robot for Dioxin/PCB Analysis, *Organohalogen Compounds*, 66, 164-168