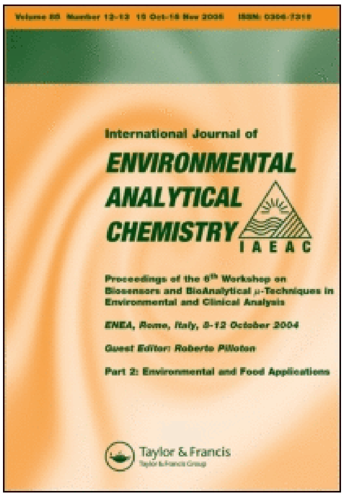

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P. Henschel[a]; H. Keune[a]; R. Kressner[a]; T. Möhlmann[b]; R. Schwabe[b]; M. Sonneborn[b]
[a] Umweltbundesamt, Berlin 33, West Germany [b] Institute for Water, Soil and Air Hygiene of the Federal Health Office, Berlin 33, West Germany

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Determination of Organic Halogen and Sulphur Compounds in Domestic Refuse Samples by Ion Chromatography[†]

P. HENSCHEL, H. KEUNE and R. KRESSNER
Umweltbundesamt, Bismarckplatz 1, D-1000 Berlin 33, West Germany

and

T. MÖHLMANN, R. SCHWABE and M. SONNEBORN
Institute for Water, Soil and Air Hygiene of the Federal Health Office, Corrensplatz 1, D-1000 Berlin 33, West Germany

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The aim of this investigation is the estimation of the impact on the environment caused by organic halogen and sulphur compounds contained in domestic refuse samples. Samples were collected in the whole Federal Republic of Germany under statistical aspects. After sorting, grinding and Wickbold-combustion the compounds chloride, fluoride and sulphate were determined.

KEY WORDS: Organohalides, organic sulphur compounds, Wickbold-combustion, Ion Chromatography, ionspecific electrode.

INTRODUCTION

There is an ever increasing demand to carry out investigations of domestic refuse not only in order to judge the possibilities for handling and elimination, but also to accomplish comprehensive physical and chemical measurements. This would represent a

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[†] Presented at the "Second Workshop on Ion Chromatography", Amsterdam, April 1982.

possibility to get basis data of many parameters to a greater extent enabling the estimation of the possible impact on the environment caused by the pollutants contained in domestic refuse.

PROCEDURE

During an investigation domestic refuse samples were taken in the whole Federal Republic of Germany under statistical aspects with the aim of obtaining a survey concerning the impact on the environment caused by domestic refuse. The contents of organically bound chloride, fluoride and sulphate in the samples were determined.

Figure 1 shows the scheme of the procedure. After sorting, sample partition and grinding the samples were ready for combustion in a Wickbold apparatus (Heraeus 4 with BITC-Burner). For the determination of the generated combustion products fluoride, chloride and sulphate, collected in bidest. water, Ion Chromatography was chosen.[1-6] This determination was carried out

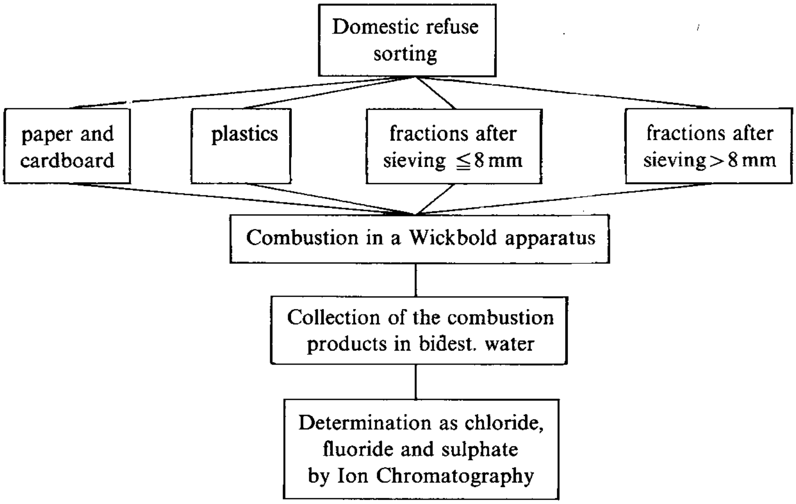

FIGURE 1. Preparation and investigation of domestic refuse samples.

by a DIONEX D 12 Ion Chromatograph with a sample loop of 100 μl.

To make this procedure of determination clear one sample is taken as an example: Figure 1 shows four different refuse fractions. From each fraction four samples were taken and combusted in the Wickbold apparatus. In the resulting sixteen aquatic solutions the determination of the compounds chloride, fluoride and sulphate was carried out four times for each sample. The results for chloride and sulphate are shown in Table I and Table II.

RESULTS AND DISCUSSION

The determination of the compounds sulphate, chloride and fluoride in domestic refuse samples after combustion produces good results by Ion Chromatography. Larger deviations between the samples of

TABLE I

Determination of organic chlorine (as Cl^-) in different fractions of domestic refuse demonstrated with a sample from an urban area. S = standard deviation; S% = standard deviation in % [ppm].

	$\bar{x}_1$	S	S%	$\bar{x}_2$	S	S%
Paper and cardboard	3.81	±0.037	0.98	3.94	±0.39	9.9
	4.50	±0.026	0.58			
	3.57	±0.041	1.15			
	3.89	±0.165	4.24			
Plastics	20.99	±0.212	1.01	21.91	±0.73	3.3
	22.43	±0.560	2.50			
	21.65	±0.144	0.67			
	22.56	±0.183	0.81			
Fractions after sieving ≦ 8 mm	0.58	±0.017	2.93	0.66	±0.18	27.3
	0.57	±0.010	1.75			
	0.55	±0.056	10.18			
	0.92	±0.022	2.39			
Fractions after sieving > 8 mm (vegetable part of domestic refuse	1.15	±0.028	2.43	1.28	±0.15	11.7
	1.42	±0.026	1.83			
	1.39	±0.018	1.29			
	1.14	±0.031	2.72			

$\bar{x}_1$:mean value of four IC—measurements of the same sample.
$\bar{x}_2$:mean value of $\bar{x}_1$.

TABLE II

Determination of organic sulphur (as SO_4^{--}) in different fractions of domestic refuse demonstrated with a sample from urban area. S = standard deviation; $S\%$ = standard deviation in % [ppm].

	$\bar{x}_1$	S	$S\%$	$\bar{x}_2$	S	$S\%$
Paper and cardboard	2.17	±0.093	4.39	2.21	±0.09	4.0
	2.11	±0.018	0.87			
	2.23	±0.022	1.0			
	2.32	±0.036	1.55			
Plastics	4.86	±0.057	1.16	4.96	±0.095	1.92
	5.09	±0.029	0.56			
	4.94	±0.049	0.98			
	4.96	±0.040	0.81			
Fractions after sieving ≤ 8 mm	0.62	±0.04	6.5	0.72	±0.106	14.7
	0.69	±0.096	1.39			
	0.87	±0.219	25.13			
	0.70	±0.013	1.86			
Fractions after sieving > 8 mm (vegetable part of domestic refuse)	2.36	±0.062	2.65	2.25	±0.074	3.27
	2.22	±0.097	4.40			
	2.21	±0.036	1.63			
	2.21	±0.010	0.43			

$\bar{x}_1$: mean values of four measurements of the same sample.
$\bar{x}_2$: mean value of $\bar{x}_1$.

one investigated refuse fraction are not caused by the manner of combustion or by the manner of detection, but by the inhomogeneity of the investigated material.

For the examination of the method PVC-powder with known content of organically bound chloride was investigated by the same method. The results in Table III show that there is a consistent agreement between the known content and the determined content (>95% of agreement) and an acceptable standard deviation of <1%.

Under the conditions of the Wickbold-combustion the generation of carboxylic acids is possible. These carboxylic acids can disturb the determination of fluoride, since the retention time of the peak of the carboxylic acids is very close to the retention time of the fluoride peak, hence sometimes a separation of the two peaks is not possible. For the examination of this possibility of disturbance the content of

TABLE III

Standardisation of the Wickbold Combustion and Ion Chromatography combination by analysing pure PVC. Theoretical content = 0.568 g Cl^-/1 g PVC; $\bar{x}$ = mean value; S = standard deviation; S% = standard deviation in % 1 g PVC.

Sample no. (1 g PVC)	x [mg Cl^-/g PVC]	$\bar{x}$ [mg Cl^-/g PVC]	S [mg Cl^-/g PVC]	S%	% Theorie
I	540.9 542.2 541.2 540.6	541.2	±70	0.13	95.3
II	542.7 549.6 550.2 553.9	549.1	±4.67	0.85	96.7
III	552.0 554.0 554.0 552.7	553.2	±0.99	0.18	97.4
IV	551.1 552.7 552.7 556.2	553.2	±2.15	0.39	97.4

fluoride was also determined by a fluoride sensitive electrode (Orion fluoride electrode Mod. 94-09). Figure 2 shows the results of this examination. The agreement between the values obtained by Ion Chromatography and the values obtained by the ionspecific electrode is very consistent, although the values of the determination by the electrode are always a little bit higher.

CONCLUSION

The described procedure enables the determination of organically bound chloride, fluoride and sulphur in domestic refuse samples. The combustion of the refuse samples by a Wickbold-apparatus proved to be effective. The found recovery ratios and the checkup of the results by a comparison between different analytical methods make clear that this procedure of determination is very suitable for the routine investigation of a larger series of samples.

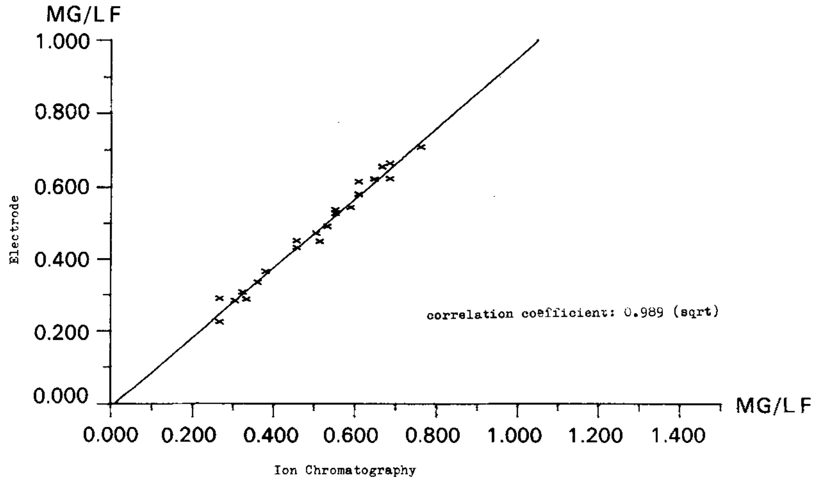

FIGURE 2. Comparison between Electrode/Ion Chromatography.

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