

Results of interlaboratory comparisons of column percolation tests

Ute Kalbe*, Wolfgang Berger, Franz-Georg Simon, Jürgen Eckardt, Gabriele Christoph

*Federal Institute for Materials Research and Testing (BAM), Division IV.3 Waste Treatment and Remedial Engineering,
Unter den Eichen 87, D-12205 Berlin, Germany*

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Abstract

Laboratory leaching tests may be used for source term determination as a basis for risk assessment for soil–groundwater pathway (leachate forecast) on contaminated sites in Germany. Interlaboratory comparisons on the evaluation of the reproducibility of column percolation tests were conducted within the framework of an integrated R + D program using three waste reference materials.

The interlaboratory comparisons of column percolation tests showed good reproducibility of the results for inorganic and organic parameters as well as for the accompanying parameters. This is due to the stipulations concerning the time of contact between leachant and sample material as well as the sample placement in the columns. Different column dimensions used by the participants of the interlaboratory comparisons did not have any substantial influence on the column test results.

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1. Introduction

The Federal Ordinance on Soil Protection and Contaminated Sites (BBodSchV) [1] requires a leachate forecast for risk assessment for the soil–groundwater pathway. Since there are considerable gaps in the practical implementation of the requirements of the BBodSchV throughout Germany, the Federal Ministry for Education and Research (BMBF) has initiated an R + D program entitled “Prediction of pollutant input into groundwater from leachate (leachate forecast)” [2]. Within the framework of this integrated program the project partners developed suitable methods for the determination of the pollutant source term. In order to ensure comparability of the methods, materials with a defined source term for certain pollutants, so-called reference materials (RM), were used. BAM (Federal Institute for Materials Research and Testing) carried out the

processing of three reference materials of about 6 tonnes each (contaminated soil, demolition waste, and municipal solid waste incineration bottom ash) [3] and the required quantities of the RMs were provided to the project partners for their specific work programs.

Comparative laboratory investigations to evaluate the reproducibility of the results of leachate forecast from several laboratory procedures (elution, extraction, and column percolation methods) were carried out [4]. The results of the interlaboratory comparison of column percolation tests are the subject of this paper.

Guidance documents regarding column percolation tests exist in several countries, e.g. in the Netherlands [5], in the USA [6], and in the Scandinavian countries [7]. Standard drafts are also available [8,9]. Whereas laboratory comparisons on leaching or extraction tests were reported within the framework of determination of pollutants from soil, waste, and other matrices [10–15], laboratory comparisons on column tests have rarely been conducted so far. Some of the few examples are the validation of NEN 7343 [5,16] and the comparison of different leaching methods [17–19]. Efforts to harmonize leaching methods have been made among others by van der Sloot et al. [20], Townsend et al. [21], and Wahlström et al. [22].

* Corresponding author. Tel.: +49 30 81043862; fax: +49 30 81041437.

E-mail addresses: ute.kalbe@bam.de (U. Kalbe),
wolfgang.berger@bam.de (W. Berger),
franz-georg.simon@bam.de (F.-G. Simon),
juergen.eckardt@bam.de (J. Eckardt),
Gabriele.christoph@bam.de (G. Christoph).

Table 1
Selected parameters of the RMs used

Parameter	Dimension	CS RM	DW RM	BA RM
Residual moisture content (DIN ISO 18121)	% by mass	0.3	2.4	3.7
pH-value (H ₂ O, DIN ISO 10390)	–	8.8	11.9	11.1
Electrical conductivity (DIN ISO 11265)	mS/cm	–	3.3	2.0
TOC (=TC-TOC) (TC DIN ISO 10694, TIC calculated from carbonate content)	% by mass	0.3	0.6	0.5
Carbonate content (Gasvolumetric)	% by mass	3.6	5.3	3.4
Loss on ignition (DIN ISO 18128)	% by mass	0.6	1.8	2.0
Particle density (He-Pycnometer)	g/cm ³	2.66	2.57	2.60
Particle size distribution (DIN 18123, wet sieving)				
4–2 mm	% by mass	–	14.9	19.5
2–0.63 mm	% by mass	33.0	29.0	26.8
0.63–0.2 mm	% by mass	60.4	36.5	31.0
0.2–0.063 mm	% by mass	5.7	17.5	17.6
<0.063 mm	% by mass	0.9	1.9	5.0
Water permeability k_f (DIN 18130)	m/s	9.3×10^{-6}	3.5×10^{-6}	1.7×10^{-6}
Sum PAH ₁₆ in solid matter (DIN ISO 13877:2000-01)	mg/kg dry matter	83.8	31.8	–
Sulfate in solid matter (DIN 40 30:1991-06)	% by mass	–	1.37	0.58
Chloride in solid matter (HNO ₃ digestion and subsequent potentiometric titration)	% by mass	–	0.09	0.27
Heavy metals (DIN ISO 11466:1995–03 (aqua regia extraction) and DIN EN ISO 11885:1998–04 (ICP OES))				
As	mg/kg dry matter	–	2.8	8.0
Pb	mg/kg dry matter	–	109	727
Cd	mg/kg dry matter	–	0.3	5.8
Cr	mg/kg dry matter	–	16.3	67
Cu	mg/kg dry matter	–	29.2	1657
Hg	mg/kg dry matter	–	<0.01	0.05
Ni	mg/kg dry matter	–	14.5	68
Zn	mg/kg dry matter	–	66	2073

2. Materials and methods

Interlaboratory comparisons were carried out on three reference materials (RMs) manufactured by BAM. The RMs used were a contaminated soil (CS RM) with the pollutant spectrum of a contaminated tar works site (grain size <2 mm), a demolition waste (DW RM) with PAH contamination including gypsum as a sulfate source (grain size <4 mm), and a municipal solid waste incineration bottom ash (BA RM) with relevant Cu and Cr contents (grain size <4 mm). Table 1 comprises a collection of parameters of the RMs used for the interlaboratory comparisons [3]. Laboratories and institutions of the integrated program “Leachate forecast” participated voluntarily in the interlaboratory comparisons after an invitation. In each case six institutes took part in the column percolation tests both on organic and inorganic parameters.

First a procedure guideline was developed in order to ensure a uniform procedure for all participants using their available column test devices. The basic conditions were selected in such a way that comparability of the results could be ensured despite the different column dimensions of the participants taking into consideration DIN CEN/TS 14405: 09.04 [8] and ISO/DIS 21268-3: 09.04 [9]. The stipulation of the contact time and the placement method for the test material in the columns provided the basis for a suitable, comparable procedure. Table 2 contains an overview of the specified parameters.

The necessary representative sample amounts were provided to the participants taking into account the respective column dimensions and the intended fill heights of the columns. This represented a quality assurance measure in order to exclude sampling errors. Two replicates (tests a and b) were performed by each participant (laboratories C1–C6). The materials were

Table 2
Overview of the specified parameters of the column percolation tests

	CS RM	DW RM	BA RM
Particle density of sample material (g/cm ³)	2.656	2.566	2.600
Bulk density of sample material in the filled-up column (g/cm ³)	1.68	1.74	1.76
Contact time eluent/sample material (h)	17	15	15

Table 3
Test conditions of the participants of the column percolation tests for the CS RM—organic parameters (sorted according to fill volume)

	Lab a	Lab b	Lab c	Lab d	Lab e	Lab f
Fill volume (cm ³)	328	391	585	674	1325	1562
Internal diameter (cm)	6.00	5.86	5.46	5.86	7.50	9.00
Fill height (cm)	11.6	14.5	25.0	25.0	30.0	24.6
Sample dry mass (g)	548	657	1047	1121	2154	2607
Bulk density (g/cm ³)	1.67	1.68	1.79	1.67	1.62	1.67
Flow rate (ml/min)	0.11	0.14	0.18	0.22	0.49	0.50
Saturation time (min)	89	120	80	102	420	213
Test time (h)	193	338	479	432	196.5	333
Contact time eluent/sample (h)	18.2	17.1	17.9	19.2	17.8	19.4

placed as delivered (air-dried) into the columns and the bulk densities achieved during placement in the column were determined precisely after placement by the participating laboratories. They were able to achieve the specified bulk densities using the specified placement method (following DIN CEN/TS 14405: 09.04) with tolerable deviations (see Tables 3 and 4). Please note that Tables 3 and 4 show the names of the laboratories a–f which are not related to the codes C1–C6 given to the laboratories. This was done to protect the laboratory anonymity. There was only one case where the prescribed placement method could not be used because of the column shape. The flow rates to be applied were determined by each laboratory based on the following calculations:

$$\text{Flow rate (ml/min)} = \frac{\text{Fill volume (ml)} \times \text{Porosity (-)}}{\text{Contact time (h)} \times 60 \text{ (min/h)}} \quad (1)$$

$$\text{Porosity (-)} = 1 - \frac{\text{Bulk density (g/cm}^3\text{)}}{\text{Particle density (g/cm}^3\text{)}} \quad (2)$$

$$\text{Bulk density (g/cm}^3\text{)} = \frac{\text{Weighed portion (g dry matter)}}{\text{Fill volume (cm}^3\text{)}} \quad (3)$$

The contact times for the experiments on the three RMs varied from 15 to 17 h (see Table 2) due to the difference in the bulk densities (see Eqs. (1) and (2)). This was necessary to ensure

simultaneous execution of the tests in columns with different RMs using one multi-channel pump based on several feasibility tests.

Before starting the tests the columns filled with the sample material were saturated rapidly. The procedure guideline suggests 10- to 15-fold of the determined flow rate. The saturation time varied considerably due to the different amounts of fill and different flow rates for saturation between 75 and 420 min. In most cases less than 3 h were sufficient. Immediately after saturation, the column percolation tests were continued with the flow rate determined for the respective test.

Since minor changes in the specified and planned test conditions can result from practical conditions of the test procedures (e.g. deviation from the placement method due to the shape of the columns, deviations in the bulk density or changes of the flow rate during execution), certain deviations from the specified conditions (Table 2) were tolerated. Tables 3 and 4 show an example of compilation of the test conditions for the column test set-ups used (sorted according to fill volume referred to as lab a–f in order to keep anonymity of the participants) for organic and inorganic parameters. Columns with internal diameters of 40–90 mm with fill heights between 12 and 30 cm were used. These dimensions suggested sample masses of between 0.5 and 3 kg and flow rates of between 0.11

Table 4
Test conditions of the participants in the column percolation tests for RM BA—inorganic parameters (sorted according to fill volume)

	Lab a	Lab b	Lab c	Lab d	Lab e	Lab f
Fill volume (cm ³)	298	391	585	674	1016	1581
Internal diameter (cm)	4.0	5.86	5.46	5.86	7.50	9.00
Fill height (cm)	23.7	14.5	25.0	25.0	23.0	24.9
Sample dry mass (g)	504	688	1041	1159	1683	2778
Bulk density (g/cm ³)	1.70	1.72	1.78	1.72	1.655	1.76
Flow rate (ml/min)	0.11	0.14	0.20	0.22	0.37	0.50
Saturation time (min)	75	90	70	88	180	215
Test time (h)	475	338	601	408	357	332
Contact time eluent/sample (h)	15.6	15.7	15.4	17.25	16.6	17.1

Table 5
Measuring program of the column percolation tests in the framework of interlaboratory comparisons

Measured parameters	CS RM	DW RM	BA RM
Investigation of inorganics			
pH value, conductivity, turbidity	X	X	X
Chloride, sulfate, Cu, Cr		X	X
Investigation of organics			
pH value, conductivity, turbidity, DOC content	X	X	
PAH	X	X	

and 0.5 ml/min when the specified contact time was adhered to. Glass columns were used, except in one case where high-grade steel columns were used. The contact time in the execution of the practical test was to some extent increased in relation to the specified time (see Tables 3 and 4). The test duration needed by the participants was within 8–25 days corresponding to L/S (liquid to solid) ratios from 3.2 to 6.7 l/kg. Sodium azide (NaN_3) was added to the eluent in order to reduce microbial activity.

The sampling plan was established by the participants individually in accordance with the specified conditions. Sampling of percolates at the beginning of the column tests was carried out more frequently. With increasing experimental time the intervals of the sampling were increased. In the column tests for the investigation of the leaching behavior of PAHs, the amount of leachate necessary for analysis had to be considered due to the detection limit of measurement. A filtration took place only through the thin filter layer (quartz sand with a 1–2 mm grain size) placed on the top of the column with a specified layer thickness (maximum 10 mm). Table 5 illustrates the measuring program to be performed on the percolates obtained. The PAHs in the percolates were measured using high-performance liquid chromatography (HPLC, 4 laboratories) and gas chromatography–mass spectrometry (GC–MS, 2 laboratories). Heavy metals were analyzed using inductively coupled plasma–optical emission spectrometry (ICP–OES, 4 laboratories) and atomic absorption spectrometry (AAS, 2 laboratories). Sulfate and chloride were determined using ion chromatography (IC) with one exception (coulometric titration).

3. Results and discussion

In contrast to batch leaching experiments, time-resolved information can be retrieved from column tests. The effluent of a column filled with contaminated material will show initially decreasing concentrations until steady-state conditions are reached. The measured concentrations in the effluent as a function of time are dependent on the terms for dispersion, advection, retardation by sorption, and for organic pollutants decay by reaction (see Eq. (4)).

$$\frac{\partial C}{\partial t} = D_L \frac{\partial^2 C}{\partial x^2} - v_x \frac{\partial C}{\partial x} - \frac{\rho}{\Theta} \frac{\partial K_d C}{\partial t} + \left(\frac{\partial C}{\partial t} \right)_{\text{rxn}} \quad (4)$$

with C concentration of the contaminant in the leachate, t time, D_L dispersion coefficient, v_x linear flow velocity, ρ bulk density of the sample in the column, Θ porosity, K_d distribution coefficient,

and index rxn indicating a chemical or biological reaction [23].

A statistical evaluation for the interlaboratory comparisons based on the column percolation tests was differing to conventional round-robin test evaluation since there were no uniform points of reference to certain L/S ratios due to individual laboratory's differences in sampling times. The optimization of the comparability of the test conditions was taken into account, rather than the concept of the statistical analysis (see Section 2). Therefore, the column percolation tests were evaluated first of all based on a graphical illustration of the test results. For the graphical evaluation, the concentration and the cumulative release of the content materials of the RM in the column percolates were plotted against the L/S ratio. Additionally, the results of the determined accompanying parameters such as DOC content, pH value, conductivity, and turbidity were also included in the evaluation.

The interlaboratory comparisons of the column percolation tests mainly showed good reproducibility both for the investigated substances and the accompanying parameters of the participating laboratories (laboratories C1–C6).

Fig. 1 shows an example of good agreement of the curves measured by the individual participants for the accompanying parameter DOC content in the column percolates of demolition waste RM.

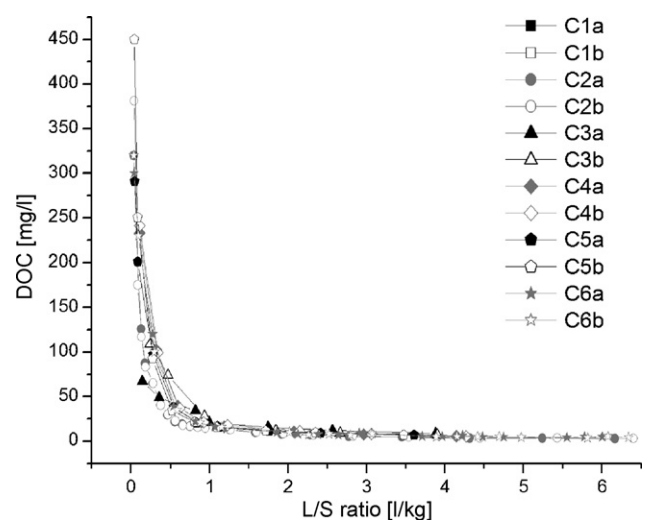


Fig. 1. Dissolved organic matter (DOC) contents of the interlaboratory comparison of column percolation tests on DW RM of participants C1–C6 (two simultaneous tests a and b).

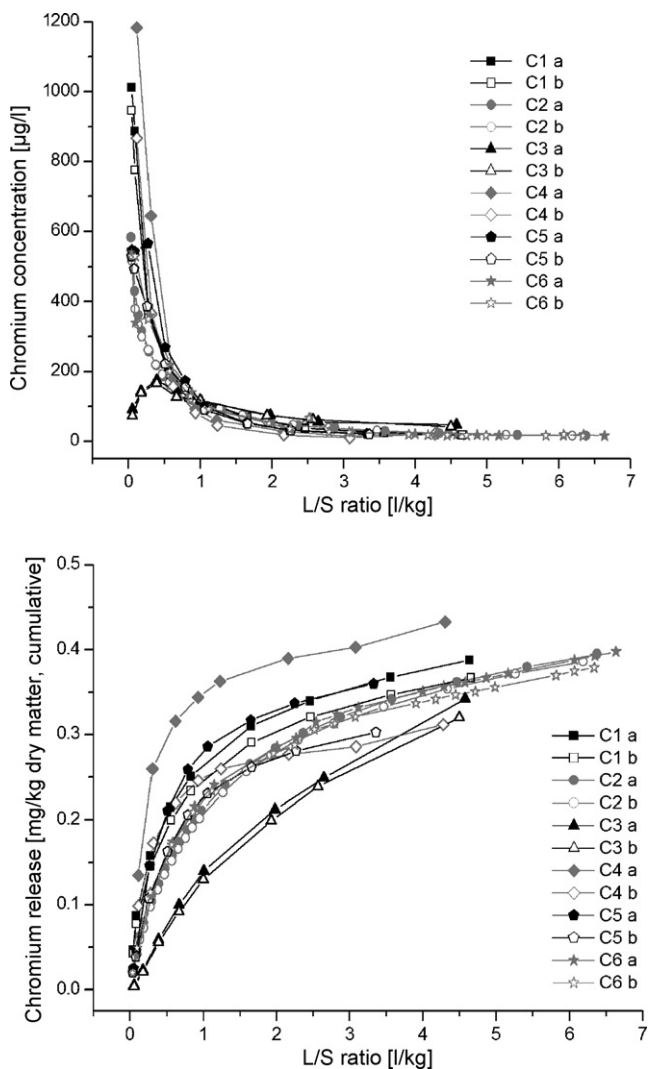


Fig. 2. Chromium concentration (top) and chromium release (bottom) of the interlaboratory comparison of column percolation tests on BA RM of participants C1–C6 (two simultaneous tests a and b).

Fig. 2 shows the good reproducibility of the determination of inorganic parameters using the example of concentration and cumulative chromium release for the BA RM. One of the laboratories (C3) clearly measured lower initial concentrations, thus the curves of Cr release from the BA RM exhibit a significantly smaller rise compared to those of other laboratories. However, starting from a certain L/S ratio, these curves also reach the same level of release determined by other laboratories. It was not possible to clarify the reason for this deviation from the test conditions. This effect occurred more or less noticeably in the same laboratory with other measured parameters as well (also see Fig. 3).

The determined initial concentrations are of crucial importance for the release of content substances (first-flush effect). In the case of the release curves measured by laboratory C4 the curve of test 4a is to be attributed firstly to the measured initial concentration. An average which includes test 4b provides a better agreement with the curves of other participants.

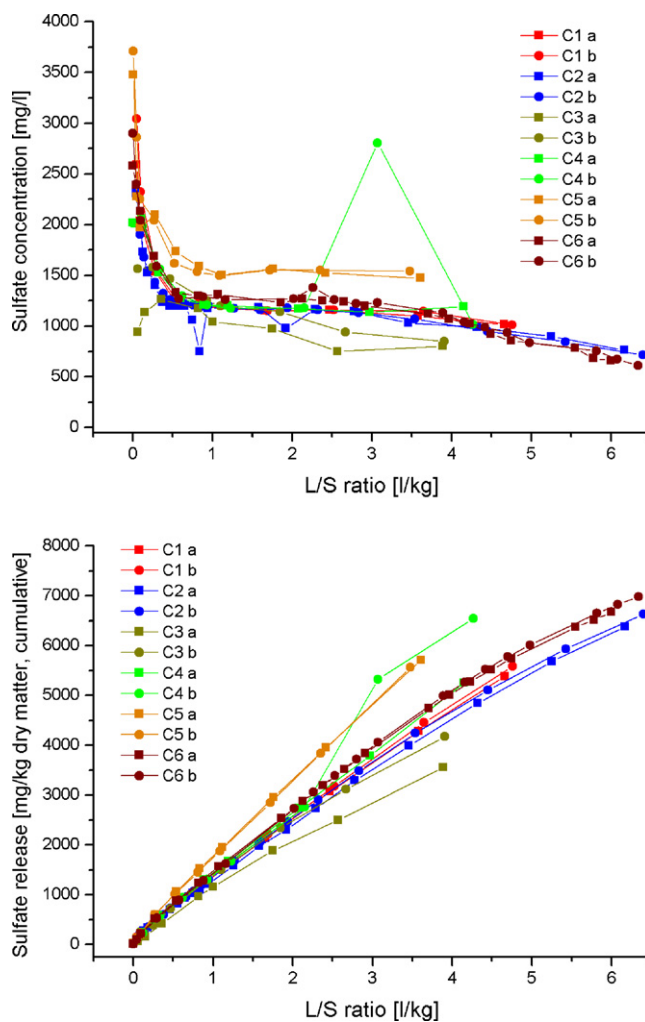


Fig. 3. Sulfate concentration and sulfate release of the interlaboratory comparison of column percolation tests on DW RM of participants C1–C6 (two simultaneous tests a and b).

The good reproducibility of the determination of anions in the column percolate is shown by the example of sulfate release from the DW RM in Fig. 3. The singular increased value in the second simultaneous test of participant C4 could be down to analytic reasons.

Figs. 1–3 indicate very good agreement of the two simultaneous tests a and b at the same time, apart for a few exceptions within the individual laboratories for the inorganic and accompanying parameters.

The column percolation tests on PAH release showed a good agreement of the results both within the laboratories (repeatability) and between laboratories (reproducibility). Only one of the participants measured substantially (several orders of magnitude) lower PAH values in the column percolates over the entire period of the tests both for soil RM and demolition waste RM. The results of this participant were considered as outliers. For the remaining participants a good agreement was found for the measured values of the 15 PAH compounds in the percolates. The release of the PAH sum is shown in Fig. 4 by the example of soil RM.

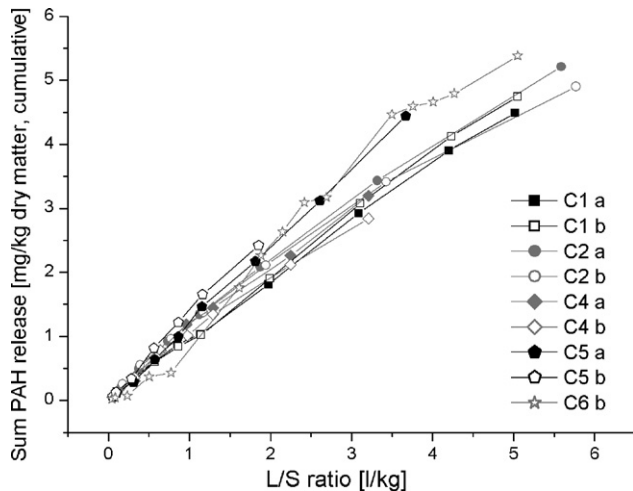


Fig. 4. Polycyclic aromatic hydrocarbon (PAH) release in the interlaboratory comparison of column percolation tests on CS RM of participants C1–C6 (two simultaneous tests a and b).

The behavior of chromium shown in Fig. 2 is different to that of PAH and sulfate shown in Figs. 3 and 4. The release of PAH in soil RM, and sulfate in demolition waste RM as a function of the L/S ratio, is almost linear because constant quantities are mobilized after initial high concentrations in the effluent. The specific release rates, i.e. the slope of the straight lines in Figs. 3 and 4, are 1270 ± 214 mg sulfate/l and 1.05 ± 0.15 mg PAH (sum)/l, respectively. The reproducibility of the measured release rates is good (standard deviation 16 and 14%, respectively) and is within the range of those reported by Hansen et al. [18]. Such a behavior is expected for contaminants with low solubilities and high total content (13.7 g sulfate/kg and 83.8 mg PAH (sum)/kg, see Table 1). Different to that is the behavior of chromium, see Fig. 2. A linear increase of the release as a function of the L/S ratio is observed only at low L/S ratios, i.e. at the beginning of the percolation test. Then the release rate decreases to a lower constant value. For the assessment, a statistical approach similar to that of Powell and Jordan [24] was selected. The slope of straight lines at L/S ratios >1 in Fig. 2b is 0.026 ± 0.005 mg Cr/l with an axis intercept of 0.25 ± 0.026 mg Cr/kg. A small amount of chromium is rapidly mobilized up to an L/S ratio of approximately 1, subsequently the release rate approaches a much lower constant value due to lower solubility but still high content (67 mg/kg, see Table 1). The behavior of sulfate in the bottom ash RM is different to that in the DW RM, (see Fig. 5) [25]. The source of sulfate in DW RM is gypsum, mainly from gypsum plasterboards, whereas sulfate in bottom ash arises from various minerals [26]. The initial slope of the straight lines in Fig. 5 is almost the same up to an L/S ratio of 0.5, where the sulfate fraction is released and can be rapidly mobilized in bottom ash RM. Then sulfate release measured approaches a lower constant value as for the heavy metal chromium (see Fig. 2). Such differences in mobilization of chemical species can only be observed in percolation tests and not in static batch tests.

The reason for a better agreement of the interlaboratory comparison results of the column percolation tests on PAH compared with the leaching tests, is the fact that the percolates were not

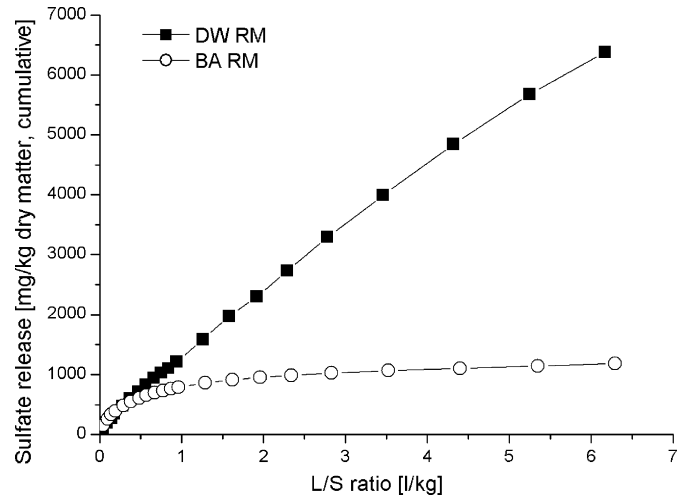


Fig. 5. Release of sulfate from DW RM and RM BA measured by laboratory C2 providing the largest number of data points.

subjected to an additional filtration. This specification ensured the avoidance of any bias by an additional sample preparation before subsequent analysis.

It was important for the comparability of the test results that the contact time between eluent and sample material was fairly uniform with only small deviations of approximately 5% on the average among the participants, and the placement method of the materials investigated was specified and kept uniform. Thus different column dimensions did not have any substantial influence on the column test results of different laboratories.

The test results including the guideline as well as the test conditions are comprehensively described in the final report of the project [6].

4. Conclusions

Comparative column percolation tests showed good reproducibility of the results for inorganic and organic parameters as well as for the accompanying parameters despite different column dimensions used by the participants. This is due to the specified uniform basic conditions concerning the time of contact eluent/sample material as well as the sample placement in the columns. From this the conclusion can be drawn that reproducible results can only be obtained by applying strict specifications for the investigational procedure. Column tests applying adequate specifications can thus be a valuable instrument for the source term determination within the framework of a leachate forecast for risk assessment of contaminated land.

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