Evaluation of various techniques for the pretreatment of sewage sludges prior to trace metal analysis by atomic absorption spectrophotometry

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Abstract

Six techniques were evaluated for their suitability for the pretreatment of dried sewage sludge prior to trace metal analysis by atomic absorption spectrophotometry. The evaluation comprised analysis of two prepared samples of dried sludge for aluminium, cadmium, chromium, copper, iron, lead, manganese, nickel and zinc, after the following pretreatment: dry ashing at 500 °C followed by extraction with dilute hydrochloric acid; dry ashing at 500 °C followed by extraction with aqua regia; nitric acid digestion followed by extraction with hydrochloric acid; extraction with aqua regia; ashing with magnesium nitrate solution at 550 °C followed by digestion with hydrochloric acid and extraction with nitric acid; extraction with nitric acid. Procedures involving the use of perchloric acid, hydrofluoric acid and hydrogen peroxide were not considered for reasons of safety. Except in the case of aluminium the direct mineral acid digestion and/or extraction methods generally gave higher recoveries than the procedures incorporating an ashing step. Direct extraction of the sample with aqua regia was recommended as a rapid and simple general method of sample pretreatment prior to analysis for all of the metals investigated except aluminium. For this metal, more drastic sample pretreatment will be required, for example fusion or hydrofluoric acid digestion.

Introduction

Industrial wastewaters discharged to the sewer often contain considerable amounts of heavy metals, which tend to become concentrated in the sludges produced during the sewage treatment process. Large quantities of these sludges are disposed to land, either for use as a fertilizer or for burial in sanitary landfills. As many of these sludges will contain trace metals in concentrations higher than those found in typical agricultural soils, their continued use could lead, in the first case, to adverse crop and food chain effects due to plant uptake of certain trace metals, and, in the second case, to groundwater contamination by toxic metals leached from the sludge. Also, certain coastal municipalities who do not have land available within a reasonable distance of their wastewater treatment plants and who do not wish to resort to expensive alternative methods of sludge treatment and disposal (such as incineration) make use of the sea as a disposal medium. This may have detrimental effects on the marine environment, in particular on accumulator organisms such as mussels and oysters (Water Research Commission, 1981). The monitoring of these sludges for heavy metal content, and consequently the necessity of having reliable methods of sample preparation, pretreatment, and analysis, has therefore become of increased importance.

The method of choice for the analysis of a sewage sludge for trace metal content would ideally be one possessing the combined attributes of accuracy, reproducibility, simplicity, speed and safety. Once sample pretreatment is complete, the most suitable and generally used method for the trace metal determination is direct flame atomic absorption spectrophotometry. It is in the sample preparation and pretreatment stages, however, where most of the problems lie, where most errors will probably occur, and where the greatest care must be taken. At the present time there is no generally recognised procedure for the treatment of a sample of sewage sludge prior to analysis for trace metals. Many acid digestion and/or extraction procedures, incorporating a wide variety of acid mixtures, some in combination with ashing or fusion techniques, have been reported, for example: nitric acid alone, hydrochloric acid alone, nitric-hydrochloric acids, nitric-sulphuric acids, nitric-perchloric acids, nitric-hydrofluoric acids, hydrochloric-hydrofluoric acids, nitric acid-hydrogen peroxide, hydrochloric acid-hydrogen peroxide, and nitric acid-hydrogen peroxide-hydrofluoric acid; ashing temperatures varied from 300 to 650 °C; lithium metaborate and sodium carbonate fusion techniques have been used (Artiola-Fortuny and Fuller, 1979; Becket, 1978; Carrondo *et al.* 1979; Delfino and Enderson, 1978; Jennis *et al.* 1980; Katz *et al.* 1981; Knechtel *et al.* 1978; Krishnamurty *et al.* 1976; Rees and Hilton, 1978; Ritter *et al.* 1978; Sommers *et al.*, 1976; Thompson and Wagstaff, 1980; Van Loon and Lichwa, 1979; Van Loon *et al.*, 1973; Van Loon and Parissis, 1969; Wiechers and Grabow, 1981).

Ideally, the acid or acid mixture used should satisfy the following criteria (Agemian and Chau, 1975):

(1) It should dissolve all metals as well as any siliceous matter present in the sample.
(2) It should destroy the organic matter present in the sample.
(3) It should not introduce any interfering ions into the sample.

Although the use of single or combined mineral acids, such as nitric and/or hydrochloric acid, can extract a high percentage of metals from the sludge (the use of sulphuric acid is not recommended because of the possible formation of insoluble sulphates), the addition of hydrofluoric acid will ensure a more effective dissolution of the silica matrix, while the addition of hydrogen peroxide or perchloric acid to the sample as well will greatly enhance the decomposition of organic matter.

The use of hydrofluoric acid, hydrogen peroxide and perchloric acid, however, presents a significant safety problem, owing to the highly corrosive nature of the hydrofluoric acid and the explosive hazards associated with hydrogen peroxide and perchloric acid. Use of these reagents is therefore not advisable in laboratories where inexperienced analysts will be carrying out the sample pretreatments and where adequate safety precautions cannot be taken.

Ashing is also an effective treatment for the removal of organic matter. The problem here lies in the choice of a temperature high enough to decompose the organic matter in the sample without at the same time causing volatilization of certain metal compounds. In general, temperatures should not exceed 500 °C.

Fusion techniques can result in the introduction of large quantities of salts into the sample solution for analysis, which can give rise to 'non-atomic absorption' or 'matrix' interference effects where the sample is being analysed by direct flame atomic absorption.

With the foregoing criteria in mind, and with the aim of avoiding, for safety reasons, the use of hydrogen peroxide, hydrofluoric acid, and perchloric acid, six techniques were selected for evaluation of their suitability for the pretreatment of dried sewage sludge prior to trace metal analysis by atomic absorption spectrophotometry.

The results obtained on two samples of dried sewage sludge, each pretreated by means of the six selected techniques, are presented in this paper.

Materials and Methods

Sample preparation

Sample No. 1 was obtained directly from the US Environmental Protection Agency (EPA). A 'Municipal Digested Sludge', it is one of a series of EPA quality control samples. The sample consisted of 55 g of freeze-dried digested sludge, which had been mixed and blended for homogeneity and then sterilized for safe handling.

Sample No. 2 was air-dried digested sludge from the Daspoort Sewage Works, Pretoria, which had been oven-dried at 105 °C, ground for 24 h in a porcelain ball-mill, sieved through a 100 mesh (0,15 mm) sieve, and mixed well for optimum homogeneity.

Table 1 gives mean values for the metals in both samples. Those for sample 1 were –

(i) obtained through exhaustive studies by EPA referee laboratories using the EPA recommended method of sample pretreatment (EPA, 1974), and were provided along with the sample; and
(ii) obtained by 20 southern African laboratories, also using the EPA recommended method of sample pretreatment, in a recent inter-laboratory comparison study (Smith, 1981).

Those for sample 2 were obtained in the same study, by:

(i) the 20 laboratories using the EPA recommended method of sample pretreatment; and
(ii) the 20 laboratories using a sample pretreatment method of their own choice.

Sample pretreatment methods

Method 1: Dry ashing at 500 °C followed by extraction with dilute hydrochloric acid – Approximately 0,5 g sample was accurately weighed into a porcelain basin and ignited at 500 °C for 2 h in a muffle furnace, then cooled. The residue was then transferred to a 100 mℓ beaker and 25 mℓ of 3 mol/ℓ hydrochloric acid added. The mixture was gently boiled on a hot-plate for 2 h, then cooled and filtered through Whatman No. 42 paper into a 100 mℓ standard volumetric flask. The filtrate was diluted to 100 mℓ with deionized distilled water (Ritter, *et al.*, 1978).

Method 2: Dry ashing at 500 °C followed by extraction with aqua regia – As for method 1 except that the residue from the ashing stage was boiled with 3 x 12 mℓ portions of aqua regia (3 parts hydrochloric acid: 1 part nitric acid) for a total of 30 min prior to filtering.

Method 3: Digestion with nitric acid, followed by extraction with hydrochloric acid – This is the technique recommended by the EPA for the pretreatment of sewage sludge samples for analysis. Approximately 0,5 g sample was accurately weighed into a 100 mℓ beaker and 3 mℓ of concentrated nitric acid were added. The beaker was placed on a hotplate and the mixture cautiously evaporated to dryness without allowing it to boil. After cooling, another 3 mℓ of nitric acid were added, the beaker covered with a watchglass, and replaced on the hotplate. The temperature was increased until a gentle reflux action was taking place. Heating was continued until the digestion was complete (indicated by a light-coloured residue). Fifteen mℓ of 1:1 hydrochloric acid were added and the beaker again gently heated for about 15 min. The watchglass and beaker walls were then washed down with deionized distilled water and the mixture filtered through Whatman No. 42 paper into a 100 mℓ standard volumetric flask. The filtrate was diluted to 100 mℓ with deionized distilled water (EPA), 1974).

Method 4: Extraction with aqua regia – Approximately 0,5 g sample was accurately weighed into a 100 mℓ beaker and 12 mℓ of aqua regia added. The beaker was covered with a watchglass and the contents heated on the medium heat of a hot-plate until all

TABLE 1
SAMPLES 1 AND 2: MEAN VALUES OBTAINED BY EPA AND SA COMPARISON STUDIES (mg/kg)

	Method			
	Sample 1		Sample 2 SA comparison study mean values	
Metal	(1) EPA mean values (EPA method)	(2) SA comparison study mean values (EPA method)	(1) Method 1 (EPA method)	(2) Method 2 (own choice)
Aluminium	4 558	4 332	10 113	13 255
Cadmium	20,8	19,1	5,8	6,0
Chromium	204	210	155	157
Copper	1 095	1 055	606	599
Iron	16 155	17 198	24 453	23 319
Lead	519	559	349	351
Manganese	205	213	459	452
Nickel	198	195	52	54
Zinc	1 323	1 367	1 782	1 794

bubbling had ceased (30 min minimum). The mixture was diluted with about 5 mℓ of deionized distilled water and filtered through Whatman No. 42 paper into a 100 mℓ standard volumetric flask. The filtrate was diluted to 100 mℓ with deionized distilled water (Van Loon and Lichwa, 1973).

Method 5: Ashing with magnesium nitrate solution, digestion with hydrchloric acid and extraction with nitric acid – Approximately 0,5 g sample was accurately weighed into a silica crucible and 5 mℓ of a 950 g/ℓ solution of magnesium nitrate added. The mixture was stirred thoroughly with a glass rod, evaporated to dryness on a water bath, then ignited at 550 °C for about 15 min. After cooling, 10 mℓ of 1:1 hydrochloric acid were added and the mixture was again evaporated to dryness. A further 5 mℓ of 1:1 hydrhochloric acid were added and the mixture again evaporated to dryness. Another 5 mℓ of 1:1 hydrochloric acid were added and the mixture washed into a 250 mℓ beaker with 50 mℓ of deionized distilled water. Ten mℓ of 50 mℓ/ℓ nitric acid solution were then added and the mixture heated on a steam bath for about 15 min, cooled and filtered through Whatman No. 42 paper into a 250 mℓ standard volumetric flask, and the filtrate diluted to 250 mℓ with deionized distilled water (Association of Official Analytical Chemists 1975; Wiechers and Grabow, 1981).

Method 6: Extraction with nitric acid, 6 h reflux – Approximately 0,5 g sample was accurately weighed into a 250 mℓ round-bottomed flask and 50 mℓ of concentrated nitric acid added. The mixture was then boiled under reflux for 6 h, after which 100 mℓ of deionized distilled water were added and the mixture was boiled for another hour. After cooling, the mixture was filtered through Whatman No. 42 paper into a 250 mℓ standard volumetric flask and the filtrate diluted to 250 mℓ with deionized distilled water (Wiechers and Grabow, 1981).

N.B. – 'Blank' determinations were carried out for each method. Analytical reagent grade chemicals were used for the preparation of all reagents.

Determination of trace metals

A Varian-Techtron Model AA5 atomic absorption spectrophotometer was used to determine, by the direct flame technique, the following trace metals in the sample extracts: aluminium, cadmium, chromium, copper, iron, lead, manganese, nickel, zinc. A cesium-lanthanum interference suppressant solution was added to each sample extract and standard solution to give a final concentration of 0,5 g/ℓ cesium and 2 g/ℓ lanthanum. All sample extracts were analysed at the same time, under the same operating conditions.

Results

Details of the results obtained from the analysis of extracts of both samples prepared by the various pretreatment methods described are given in Tables 2 and 3. Results presented are the average values of triplicate determinations.

The results are presented graphically for comparison in Figures 1 to 3.

TABLE 2
Sample 1: (EPA MUNICIPAL DRIED SLUDGE) – ANALYSIS RESULTS (mg/kg)

	Method					
Metal	1 Dry ashing – 500 °C Extraction with 3 mol/ℓ HCl	2 Dry ashing – 500 °C Extraction with aqua regia	3 HNO_3 digestion Extraction with HCl	4 Extraction with aqua regia	5 Ashing with $Mg(NO_3)_2$ – 550 °C, HCl digestion. Extraction with HNO_3	6 Extraction with HNO_3 (6 h reflux)
Aluminium	5 900	5 800	4 400	4 200	5 200	4 400
Cadmium	17	17	18	18	17	20
Chromium	189	190	200	202	195	200
Copper	984	980	1 046	1 056	965	990
Iron	16 200	16 000	16 400	16 000	16 100	16 600
Lead	500	500	546	540	535	535
Manganese	210	212	216	218	210	220
Nickel	184	183	190	191	185	190
Zinc	1 240	1 240	1 300	1 310	1 100	1 350

TABLE 3
Sample 2: (PRETORIA MUNICIPAL DRIED SLUDGE) – ANALYSIS RESULTS (mg/kg)

	Method					
Metal	1 Dry ashing – 500 °C Extraction with 3 mol/ℓ HCl	2 Dry ashing – 500 °C Extraction with aqua regia	3 HNO_3 digestion Extraction with HCl	4 Extraction with aqua regia	5 Ashing with $Mg(NO_3)_2$ – 550 °C, HCl digestion. Extraction with HNO_3	6 Extraction with HNO_3 (6 h reflux)
Aluminium	18 600	18 000	12 000	11 000	14 500	11 400
Cadmium	6	6	6	6	6	8
Chromium	140	141	150	149	143	145
Copper	586	586	594	594	535	555
Iron	24 600	24 600	24 000	23 800	23 900	24 500
Lead	338	324	364	340	322	310
Manganese	452	460	462	464	450	460
Nickel	52	61	60	61	55	60
Zinc	1 740	1 740	1 780	1 790	1 500	1 850

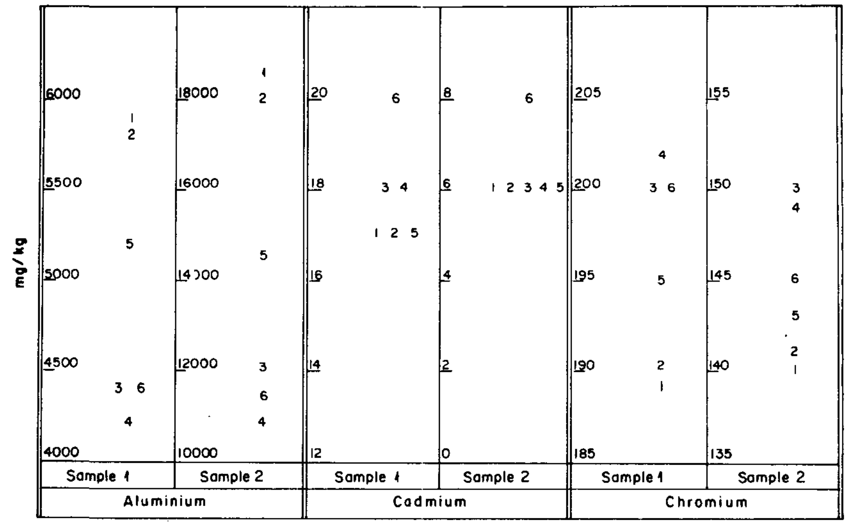

Figure 1
Comparison of aluminium, cadmium and chromium results obtained by methods 1 – 6

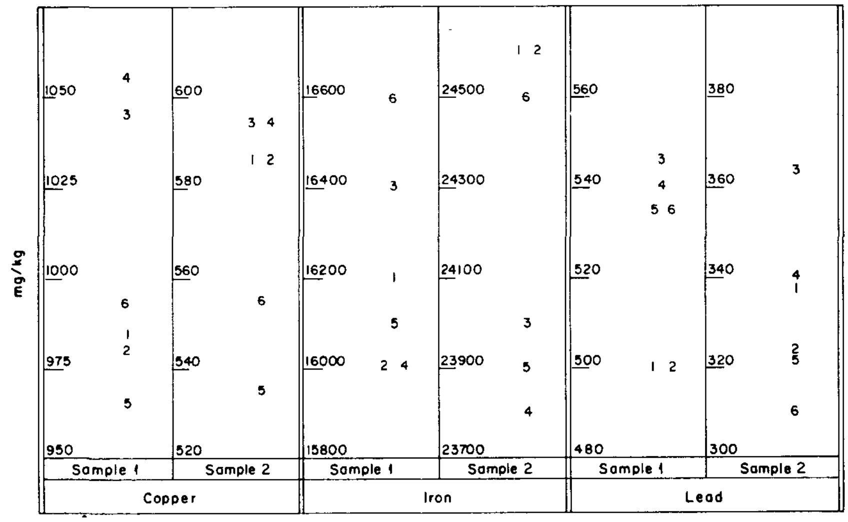

Figure 2
Comparison of copper, iron and lead results obtained by methods 1 to 6

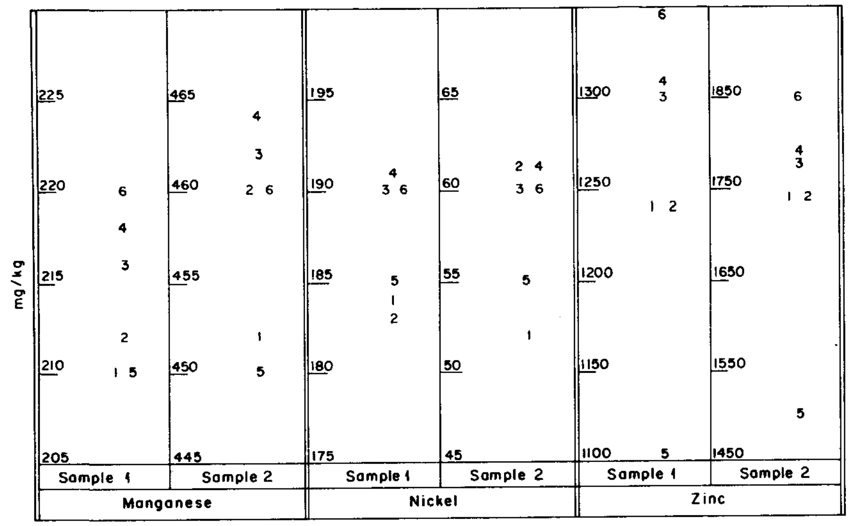

Figure 3
Comparison of manganese, nickel and zinc results obtained by methods 1 to 6

Discussion

Determination of trace metals in the sample extracts

Wavelength: The recognised standard wavelengths (EPA, 1979; *Standard Methods,* 1975) were used for all metals except lead, where the 217,0 nm wavelength was preferred. Sample extracts were diluted where necessary.

It is common practice to use a less sensitive wavelength where high concentrations of a metal are present, instead of diluting the sample solution. In many cases, however, dilution can result in a considerable reduction in interference effects, and should, in these circumstances, be the preferred technique.

Flame: The air-acetylene flame was used for the determination of all metals except aluminium and chromium; for these metals the nitrous oxide-acetylene flame was selected. The use of the air-acetylene flame for the atomic absorption determination of cadmium, copper, iron, lead, manganese, nickel and zinc, and of the nitrous oxide-acetylene flame for the determination of aluminium, is stipulated in both *Standard Methods* (1975) and the EPA (1979) manual. In the case of chromium, however, the former manual recommends the air-acetylene flame and the latter the nitrous oxide-acetylene flame. Use of both flames in this investigation showed that the determination of chromium in the nitrous oxide-acetylene flame gave higher results than were obtained in the air-acetylene flame, even with the addition of interference suppressants and use of a reducing (fuel-rich) flame. The nitrous oxide-acetylene flame is therefore preferred for the direct flame atomic absorption determination of chromium in sewage sludges.

Interference suppression: A cesium/lanthanum interference suppressant solution was added to each sample extract and standard solution in order to reduce chemical and ionisation interferences. The use of suitable interference suppressants is considered advisable with samples of this type, particularly for the determination of chromium, iron and manganese.

Background correction: The instrument used in this investigation was not equipped with background correction facilities. For greater accuracy, background correction should be applied where necessary in samples of this type. Knechtel *et al.* (1978) recommend background correction in the determination of cadmium, lead and nickel in particular, and especially for sludges high in calcium (Table 4).

Evaluation of sample pretreatment methods

Aluminium: For both samples, considerably higher results were obained using pretreatment techniques incorporating an ashing stage (methods 1 and 2) than were obtained using direct acid digestion or extraction procedures, or both. Very much higher results for the aluminium content of sample 2 were obtained in a recent interlaboratory comparison study involving this sample (Smith, 1981) by two laboratories employing fusion techniques and by one laboratory using a hydrofluoric acid-perchloric acid-hydrochloric acid digestion pretreatment procedure. Results of 35 600, 32 700 and 31 200 mg/kg Al were obtained.

Therefore, for the more accurate determination of aluminium in sewage sludges, more drastic sample pretreatment procedures, such as fusion or hydrofluoric acid digestion, are necessary in order to dissolve the aluminium present in the form of silicates and possibly other relatively insoluble matrices. Cognisance should, however, be taken of the inherent problems involved in the use of both these techniques.

Cadmium: Only a few mg/kg of cadmium were present in each sample. The results obtained by each method were almost identical, with the exception of those from method 6 (nitric acid extraction), which were slightly higher.

Iron: The differences among the results obtained for this metal by the various pretreatment methods were not considered significant.

Chromium, copper, lead, manganese, nickel and zinc: Results for these metals showed that, in general, the highest recoveries were obtained using methods 3 (nitric acid digestion—hydrochloric acid extraction), and 4 (aqua regia extraction) and, to a lesser extent, method 6 (nitric acid extraction). The lowest values were obtained using the three techniques involving an ashing step, indicating that even at 500 °C some volatilization of these metals occurred. These findings are in contrast to those reported by Ritter *et al.* (1978), who found dry ashing at 500 °C to be the most suitable of five techniques investigated for the pretreatment of sludge samples for the determination of cadmium, copper, lead, nickel and zinc, but in agreement with those of Katz *et al.* (1981), who obtained low recoveries with dry ashing pretreatment procedures in comparison with various acid digestion techniques. Thompson and Wagstaff (1980) found nitric acid digestion to give the best results compared to six other pretreatment techniques, including dry ashing at 420 and 600 °C.

TABLE 4
EFFECT OF APPLICATION OF BACKGROUND CORRECTION ON CADMIUM, LEAD AND NICKEL DETERMINATIONS ON SEWAGE SLUDGES (KNECHTEL *et al.*, 1978)

	Sample No.							
	1		2		3		4	
Metal	With correction	Without correction	With correction	Without correction	With correction	Without correction	With correction	Without correction
Cadmium, mg/kg	18,7	2,4	88,6	90,2	5,3	7,8	2,1	6,0
Lead, mg/kg	1042	1082	3090	3350	557	588	101	136
Nickel, mg/kg	-	-	-	-	-	-	6,1	26,5
Calcium, g/kg	45		49		133		273	

Sampling and sample preparation

Because of the heterogeneous nature of sludges, sampling and sample preparation prior to pretreatment and analysis should be carried out with extreme care if the sample is to be a truly representative one.

For wet sludges, the sample should be analysed as soon as possible. Where possible, it should be maintained at 0 to 5 °C or suitable preservatives added, or both, depending on the constituent or parameter being determined. In the case of air-dried sludges, it is recommended that a composite sample be prepared from about 20 to 25 'cores', collected from throughout the bulk. Subsampling of this composite and subsequent drying, grinding and sieving procedures should be aimed at producing a truly homogeneous sample which can pass through a 0,5 mm sieve (HMSO, 1977).

Conclusions

As previously stated, the use of hydrofluoric acid for the dissolution of siliceous matter and of hydrogen peroxide and perchloric acid for the decomposition of organic matter was not considered for this investigation, for safety reasons. Dry ashing, another technique for organic matter removal, was found to give low recoveries for certain metals.

Nevertheless, with the exception of aluminium, good recoveries were obtained using mineral acid digestion and/or extraction pretreatment procedures. The EPA recommended method of digestion with nitric acid, followed by extraction with hydrochloric acid, and the more rapid and simple aqua regia extraction method of Van Loon and Lichwa (1973) gave similar recovery values for the other eight metals investigated.

For general use, therefore, and for reasons of safety, simplicity, speed and reliability, the direct aqua regia extraction method is recommended for the pretreatment of sewage sludges prior to analysis for these metals.

If an accurate assessment of the aluminium content is required, more drastic sample pretreatment, in the form of fusion or hydrofluoric acid digestion, will be necessary, in which case adequate safety precautions should be taken.

Alternative methods of sample pretreatment are also advisable for the determination of more volatile trace metals such as mercury and arsenic.

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