Importance, determination and occurrence of inorganic chemical contaminants and nutrients in South African municipal sewage sludges†

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Abstract

Twelve major inorganic chemical contaminants were identified as being commonly present in municipal sewage sludges and suitable analytical methods were developed and tested for their determination, as well as for the determination of the major nutrients commonly present in such sludges. Samples of air-dried sludge were collected from seventy-seven South African sewage works and analysed for both inorganic chemical contaminant and nutrient content. The results obtained provided valuable information on the chemical quality of South African sewage sludges. In addition, they were used, in conjunction with previous related work conducted by the National Institute for Water Research (CSIR) and with reference to limits laid down by various overseas countries, to prepare a set of suggested guidelines for the application of disinfected sewage sludge to agricultural land in this country.

Introduction

The application of sewage sludge to agricultural land, apart from often being the least expensive alternative, can also be beneficial in that, in addition to its soil-conditioning properties, the sludge also contains significant amounts of the major nutrients nitrogen, phosphorus and, to a lesser extent, potassium, as well as minor nutrients such as calcium, magnesium, and certain trace elements.

However, sewage sludges also contain toxic substances in concentrations higher than those found in typical soils, and their continued application could lead to adverse crop and food chain effects, due to plant uptake of certain metals and to other contaminants which may be present in the sludge, not only in both organic and inorganic chemical forms, but also in the form of pathogens. Therefore, it is essential that sludges destined for application to agricultural land are disinfected (Oberholster, 1983), in order to eliminate any pathogenic organisms present, and also adequately monitored for the presence of organic and inorganic chemical contaminants. In this regard, countries such as the UK, USA and Canada have carried out countrywide chemical analyses of their sewage sludges, and, based on the results obtained, have subsequently established suitable guidelines for the application of this material to agricultural land.

Since the chemical nature of sewage sludge varies widely, and sludges in many parts of South Africa may not be contaminated with hazardous compounds to the same extent as highly industrialised countries abroad, the National Institute for Water Research (now the Division of Water Technology) of the CSIR undertook to carry out a similar exercise in this country, rather than merely adapt overseas guidelines. It was also envisaged that the analysis results obtained would provide useful "baseline" information on the concentrations of the various contaminants and nutrients present in South African sewage sludges, and would assist in assessing the suitability of individual municipal sludges for agricultural and horticultural applications. The results of the work, carried out between 1982 and 1989, on the inorganic chemical characterisation of South African municipal sewage sludges, are presented in this paper (Smith and Vasiloudis, 1990). A parallel project on the organic chemical contaminants found in sewage sludges was also completed in 1989 (Van Steenderen, 1989).

Importance

Increased yields from the application of sewage sludge to agricultural land come mainly from the nutrients nitrogen and phosphorus contained in the sludge. Other inorganic chemical components of sewage sludge, such as potassium, magnesium, calcium and certain trace elements are also beneficial. Only a small amount of potassium is available in sewage sludge at normal application rates and additional amounts of this element are necessary for crops with a high potassium requirement. Magnesium has a minor nutritional effect. In sludges to which lime has been added as a stabiliser or conditioner, the calcium present will help in correcting acidity in soils of low pH value.

Twelve major inorganic chemical contaminants (cadmium, chromium, copper, lead, nickel, zinc, molybdenum, mercury, arsenic, selenium, boron and fluoride) were identified as being commonly present in sewage sludge. Some of these, for example copper, zinc, boron, selenium and molybdenum, are, in low concentrations, essential to the growth of plants, and may be regarded as trace nutrients. When they are present in higher concentrations, however, adverse effects can occur, viz:

- phytotoxicity, in which crop growth and productivity are reduced; and
- elements may be taken up into the edible parts of crop plants and enter the food chain of animals and humans, with possible deleterious effects.

Other elements, such as arsenic, cadmium, lead, nickel and mercury, appear to have no beneficial effects. Specific characteristics of the various contaminants, as well as those of the pH, which can also play a significant role, are listed in Table 1 (Department of the Environment, 1977; 1981a,b; Davis, 1980; Manual of British Practice in Water Pollution Control, 1978).

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TABLE 1 POTENTIAL ADVERSE EFFECTS OF MAJOR INORGANIC CHEMICAL CONTAMINANTS IN SEWAGE SLUDGE	
Contaminant	**Potential adverse effects**
Zinc	Phytotoxic in excess. Low toxicity to animals and humans. Readily translocated.
Copper	Phytotoxic in excess. Toxic in excess to animals. Limited translocation.
Nickel	Phytotoxic. Low toxicity to animals. Limited translocation.
Cadmium	Phytotoxic. Highly toxic to animals and humans. Readily translocated.
Lead	Not very phytotoxic. Highly toxic to animals and humans. Main risk lies in ingestions by grazing animals.
Chromium	Normally present in relatively non-toxic trivalent form.
Mercury	Not very phytotoxic. Highly toxic to animals and humans.
Molybdenum	Not phytotoxic. High intakes in grazing animals can cause copper deficiency problems.
Arsenic	Plant growth can be affected if 'available' arsenic >5 mg/kg.
Selenium	Toxic to animals in excess. In alkaline soils a selenium content of > 5 mg/kg will have adverse effects.
Boron	Phytotoxic in excess. Not toxic to animals at levels normally found in plants.
Fluoride	Taken up by plant roots to limited extent. Excess can cause fluorosis in grazing animals.
pH	Availability of molybdenum and selenium increases with increasing soil pH. Aluminium and manganese can be toxic in very low pH soils.

Determination

A literature survey, carried out in 1982, of the analytical methods then available for the determination of inorganic chemical contaminants and nutrients in sewage sludge, highlighted a significant lack of acceptable standard methods for this purpose. Subsequent to this survey, however, the UK Department of the Environment, published, at intervals, several recommended methods for the analysis of many of the major inorganic chemical contaminants and nutrients, and several of these were investigated and either adapted or incorporated in the methods eventually used for their determination (Smith, 1984a, 1989). These methods, with the exception of those for arsenic, selenium, molybdenum, boron and fluoride, were further tested by carrying out an interlaboratory comparison study involving the measurement of the relevant determinands in a sample of dried sewage sludge by 29 laboratories using the proposed procedures (Smith, 1984b). Methods for arsenic, selenium, boron, molybdenum and fluoride were later investigated and evaluated. All of the analysis methods were chosen for their suitability in terms of safety, simplicity and reliability. In this regard, the use of such hazardous chemicals as hydrofluoric acid, hydrogen peroxide, and perchloric acid for the pretreatment of the sludge samples prior to analysis was avoided, while the use of instrumental analytical techniques was limited to potentiometric, UV/visible and atomic absorption spectrophotometric measurements.

A brief summary of the analytical techniques employed for the sewage sludge analysis is given in Table 2 (Standard Methods, 1980; Department of the Environment, 1978a, b, 1981c, 1982, 1983a, b, 1986, 1987; Smith, 1983, 1984a, 1989; Environmental Protection Agency, undated; Parker, 1972; Yorkshire Water Authority, 1981; Rea, 1979; Allen, 1974; Martin and Kopp, 1975; Hall and Gupta, 1969; Bye *et al.,* 1983).

Occurrence

Seventy-seven sewage works throughout South Africa, in most cases those serving populations of over 25 000, were contacted by letter soliciting their cooperation in the collection of samples of air-dried sludge from their works. A recommended sample collection procedure (Department of the Environment, 1978c) was supplied to each participating works. During the period between June 1983 and March 1987, samples of air-dried sludge were collected, at 3-monthly intervals for a period of one year, from each of 9 sewage works in the Orange Free State, 33 in the Transvaal, 7 in Natal, and 28 in the Cape Province. The samples were submitted to the National Institute for Water Research for analysis for the presence of the inorganic contaminants and nutriens previously discussed by the methods summarised in Table 2. Before carrying out the analyses, the submitted samples were pretreated according to the flow diagram in Fig. 1 (The freezing step was included mainly to eliminate small insects etc.).

The average (in the case of cadmium, chromium, copper, lead, nickel, zinc, calcium, magnesium, potassium and pH) and com-

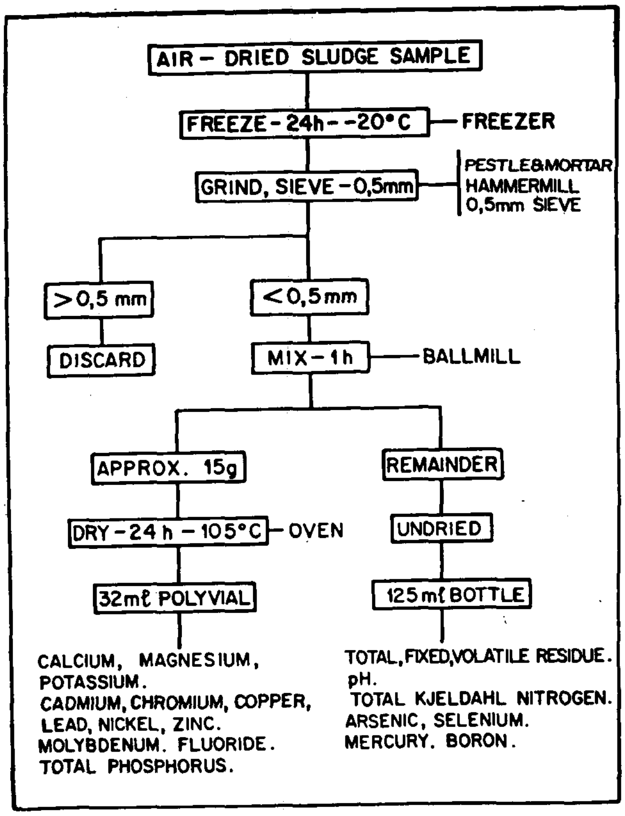

Figure 1
Flow diagram of sewage sludge sample preparation

TABLE 2
SUMMARY OF ANALYSIS TECHNIQUES

Determinand	Analysis technique
Total, volatile and fixed residue	Gravimetric: 5-10 g sample, 105°C and 550°C.
pH	Potentiometric: 5 g sample (db) → 100 mℓ.
Total Kjeldahl nitrogen	Acidimetric titration: 0,5 g sample (db); digest with sulphuric acid, potassium sulphate, mercuric sulphate; distil into boric acid; titrate with standard sulphuric acid.
Total phosphorus	Colorimetric: 0,2 g dried sample; digest with sulphuric acid and potassium persulphate, treat with sodium bisulphite; react with ammonium molybdate, potassium antimonyl tartrate and ascorbic acid; measure blue complex formed at 660 nm.
Calcium, magnesium, potassium (acid-soluble)	Atomic absorption (direct flame): 0,5 g dried sample, extract with aqua regia, add caesium-lanthanum; measure calcium and magnesium by nitrous oxide-acetylene flame and potassium by air-acetylene flame.
Cadmium, chromium, copper, lead, nickel, zinc (acid-soluble)	Atomic absorption (direct flame): 0,5 g dried sample, extract with aqua regia, add caesium-lanthanum; measure cadmium, copper, lead, nickel and zinc by air-acetylene flame and chromium by nitrous oxide-acetylene flame.
Mercury (total)	Atomic absorption (cold vapour generation): 0,5 g sample (db), digest with potassium permanganate, potassium persulphate, sulphuric acid; add sodium chloride/hydroxylamine sulphate, nitric acid, then stannous chloride; measure mercury in evolved vapour.
Arsenic and selenium (total)	Atomic absorption (hydride generation): 0,25 g sample (db), digest with nitric acid and ash with magnesium nitrate at 550°C; extract with hydrochloric acid; add sodium borohydride and measure arsenic and selenium in evolved hydrides.
Molybdenum (total)	Colorimetric: 0,5 g dried sample, ash at 500°C, extract with hydrochloric acid; add potassium thiocyanate, extract amber complex formed with MIBK and measure at 470 nm.
Boron (water-soluble)	Colorimetric: 0,2 g dried sample, extract with hot water; add azomethine-H, measure yellow complex formed at 420 nm.
Fluoride (acid-soluble)	Potentiometric: 0,5 g dried sample, extract with sulphuric acid; add ammonium hydroxide to pH 8 to 8,5; filter; measure fluoride by ion-selective electrode.

posite sample (in the case of mercury, arsenic, selenium, molybdenum, boron, fluoride, total Kjeldahl nitrogen and total phosphorus) results are summarised in Tables 3 and 4, which also show, for comparison purposes, results from similar surveys carried out in the UK (Manual of British Practice in Water Pollution Control, 1978; Davis, 1980). The "spread" of values obtained for each contaminant and nutrient is shown in the form of a histogram (Figs. 2 to 19).

Comparison of the results obtained for inorganic chemical contaminants in South African sludges with those obtained for UK sludges showed that in only three cases, i.e. copper, lead and selenium, were the maximum range values significantly higher for South African sludges than for UK sludges. UK "common" values were higher than South African median values for all contaminants (Table 3). Inorganic chemical contaminants in municipal sewage sludges can emanate from a wide variety of sources. Some of the more common of these (for the heavy metals) are listed in Table 5.

TABLE 3
SUMMARY OF CONTAMINANT CONCENTRATIONS (mg/kg, DRY BASIS) FOUND IN SOUTH AFRICAN AND UK SEWAGE SLUDGES

Contaminant	South Africa			UK	
	Range	Mean value	Median value	Range	"Common" value
Cadmium	< 1-22	12	3	2-1 500	20
Chromium	25-10 015	551	220	40-14 000	400
Copper	80-17 217	654	355	200-8 000	650
Lead	67-10 137	452	214	50-3 600	400
Nickel	6-2 660	154	55	20-5 300	100
Zinc	237-17 680	2 054	1 432	600-20 000	1 500
Mercury	< 1-22	5	3	< 1-18	5
Arsenic	< 1-34	7	6	3-30	20
Selenium	< 1-107	4	2	1-10	3
Molybdenum	1-24	6	5	1-40	6
Boron	6-78	31	28	15-1 000	30
Fluoride	32-1 260	128	97	60-40 000	250
pH (pH units)	5,4-7,8	6,8	6,8	--	--

TABLE 4
SUMMARY OF NUTRIENT CONCENTRATIONS (g/kg, DRY BASIS) FOUND IN SOUTH AFRICAN AND UK (MAINLY DIGESTED) SEWAGE SLUDGES

Nutrient	South Africa	UK
	Range	Range
Nitrogen	17-58	15-25
Phosphorus	4-41	5-18
Potassium	1-11	1-3
Calcium	11-79	16-25
Magnesium	2-13	1-5

TABLE 5
MAJOR SOURCES OF HEAVY METALS IN SEWAGE SLUDGE

Metal	Major source
Zinc	Domestic wastes. Galvanising, cosmetics, pharmaceutical and rubber industries.
Copper	Electroplating and chemical industries.
Nickel	Electroplating, motor vehicle, aircraft, printing and chemical industries.
Cadmium	Electroplating, pigment and chemical industries.
Lead	Battery, printing and paint industries. Petrol additives washed from road surfaces.
Chromium	Electroplating, tanning and dyestuff industries.
Mercury	Electrical, explosives, pharmaceutical, pesticides and chemical industries. Laboratory waste waters

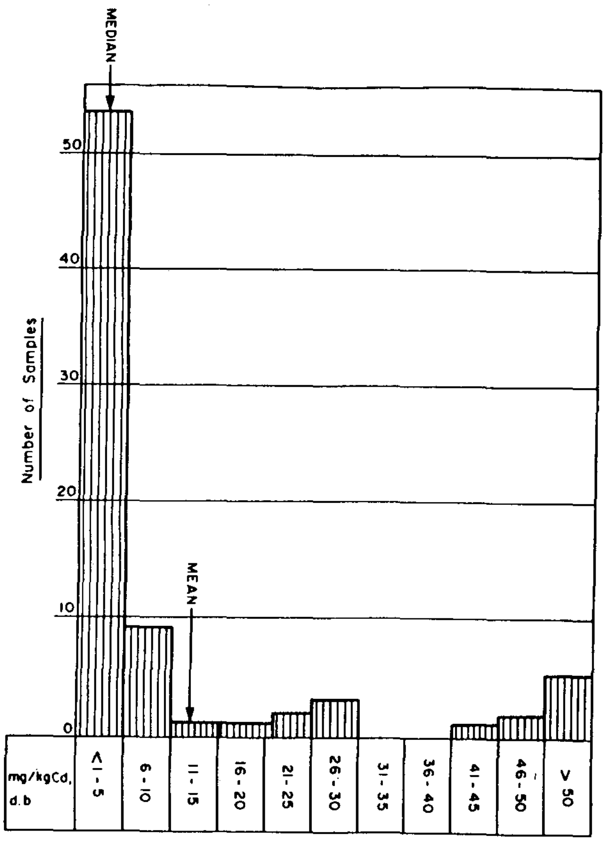

Figure 2
Cadmium concentrations in SA sewage sludges

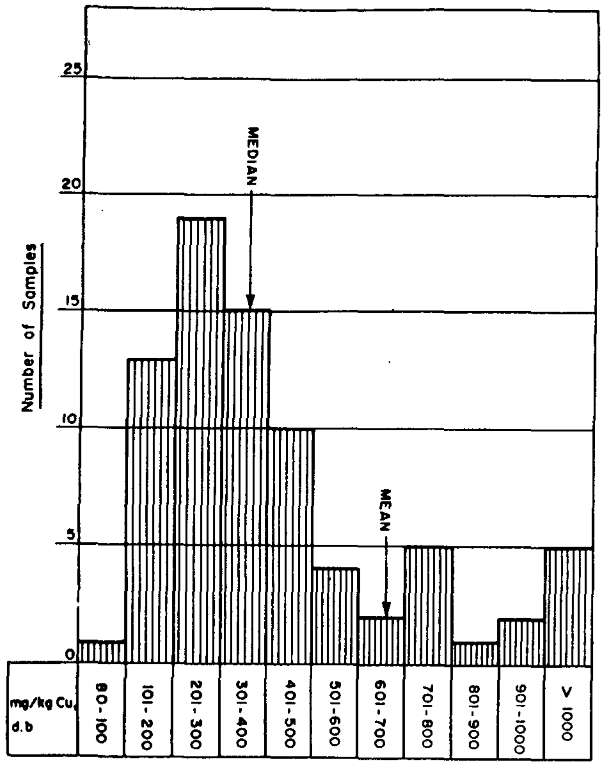

Figure 4
Copper concentrations in SA sewage sludges

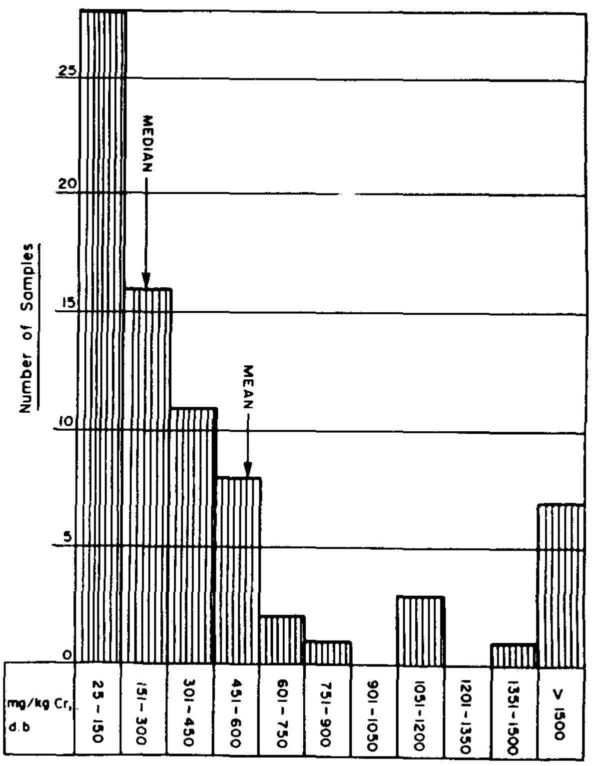

Figure 3
Chromium concentrations in SA sewage sludges

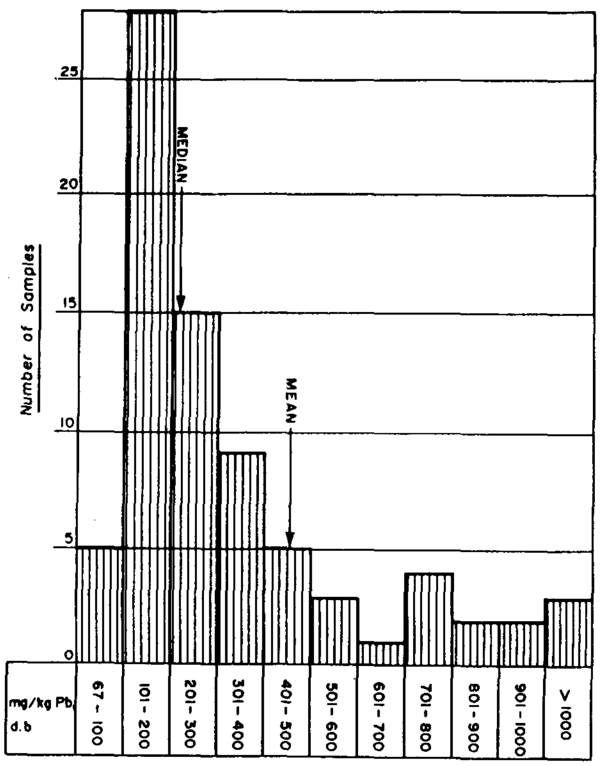

Figure 5
Lead concentrations in SA sewage sludges

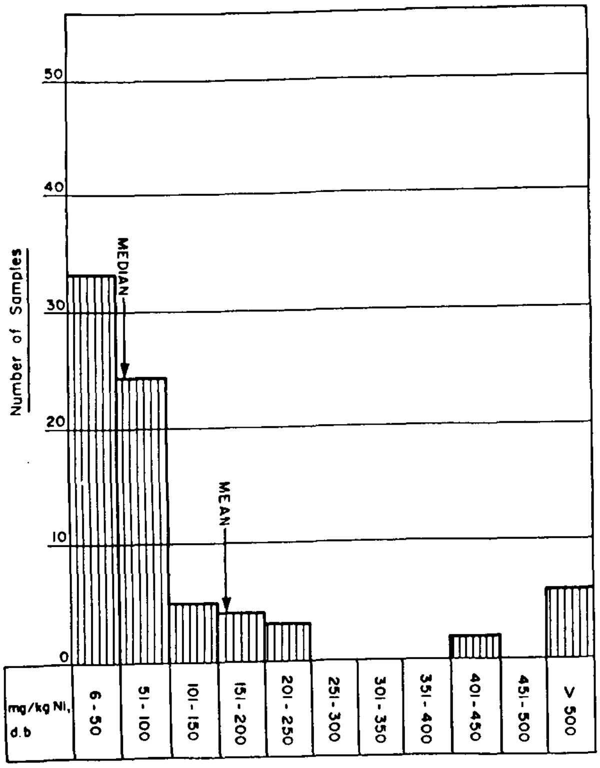

Figure 6
Nickel concentrations in SA sewage sludges

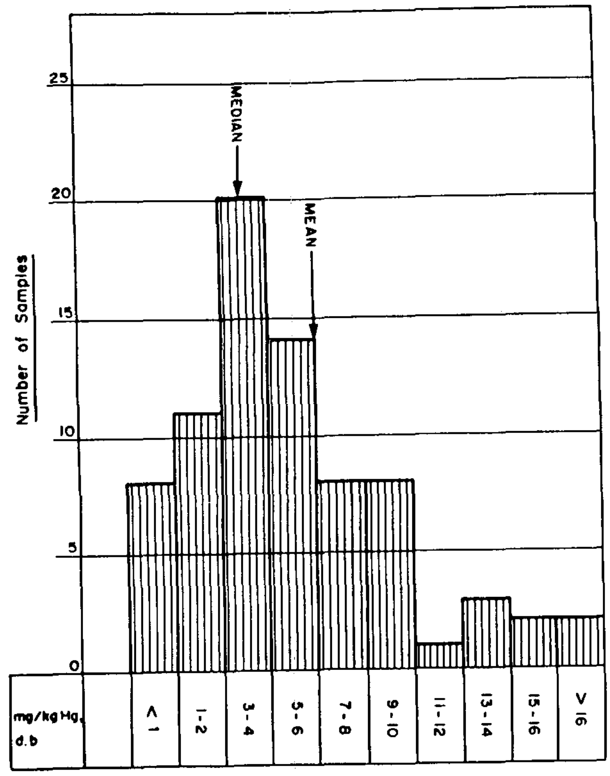

Figure 8
Mercury concentrations in SA sewage sludges

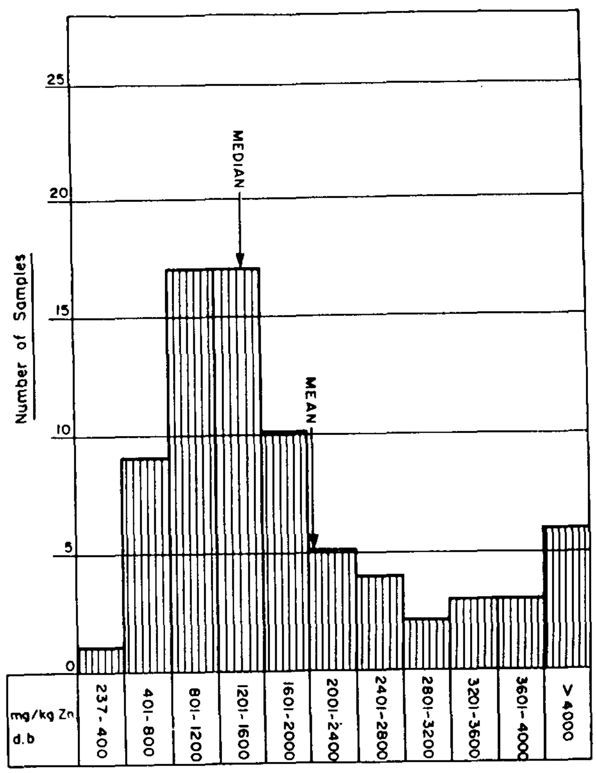

Figure 7
Zinc concentrations in SA sewage sludges

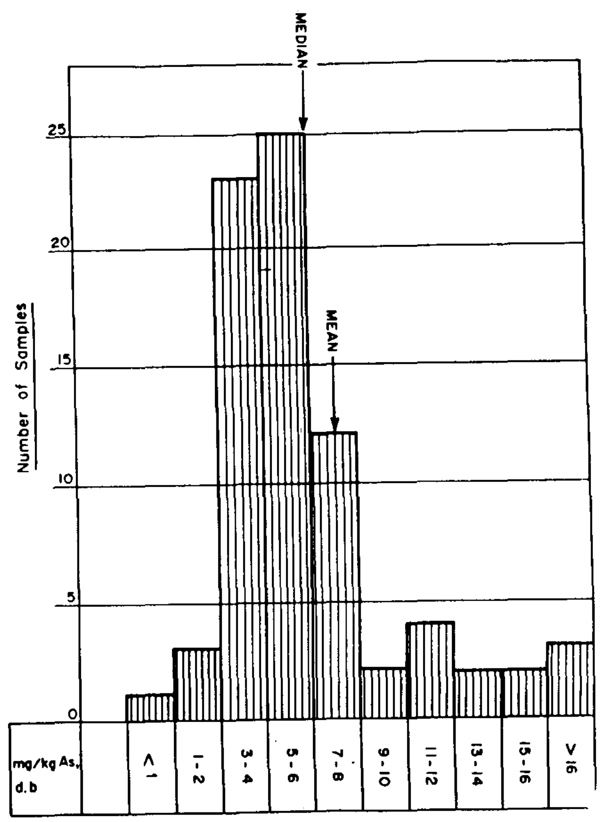

Figure 9
Arsenic concentrations in SA sewage sludges

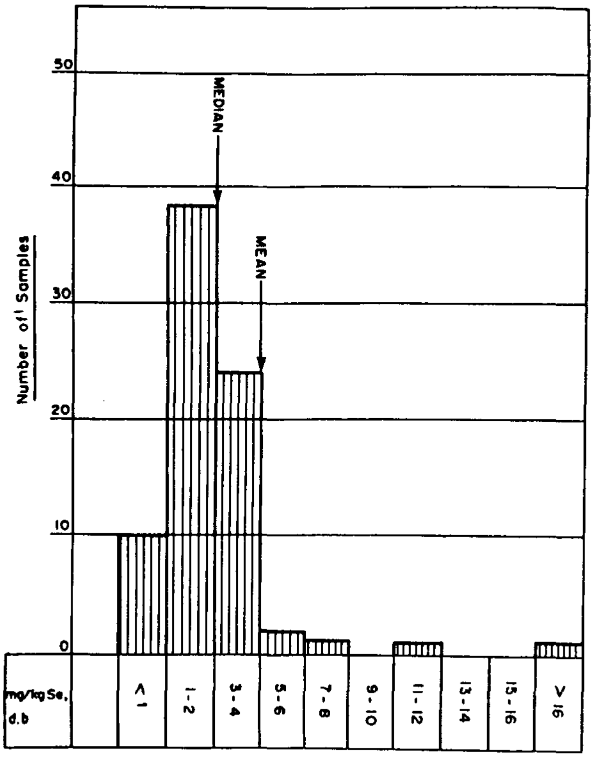

Figure 10
Selenium concentrations in SA sewage sludges

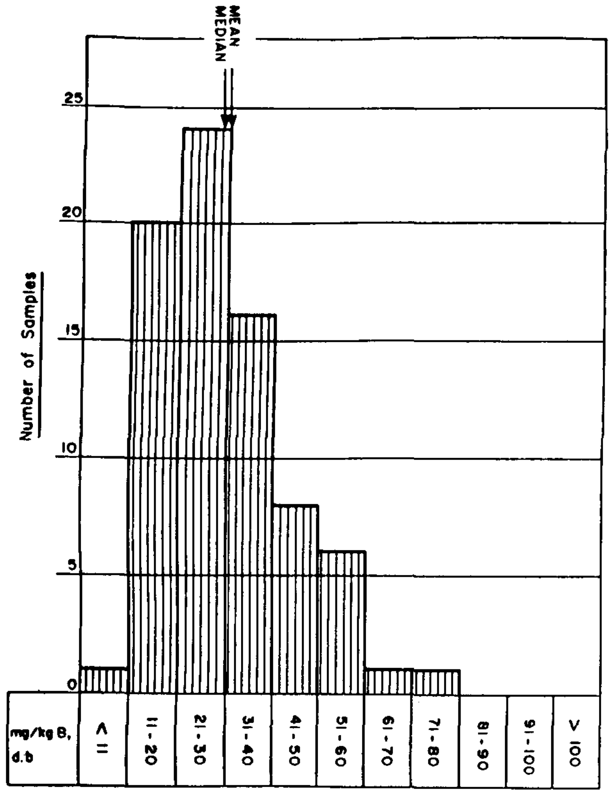

Figure 12
Boron concentrations in SA sewage sludges

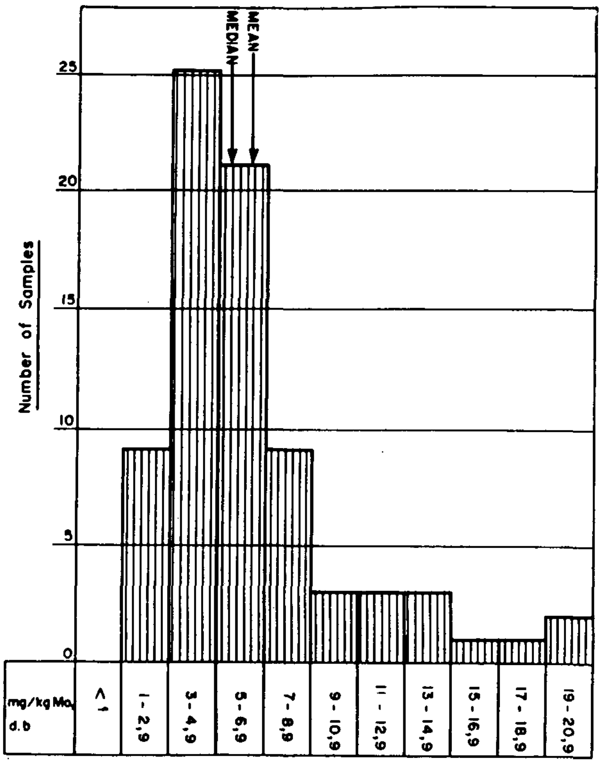

Figure 11
Molybdenum concentrations in SA sewage sludges

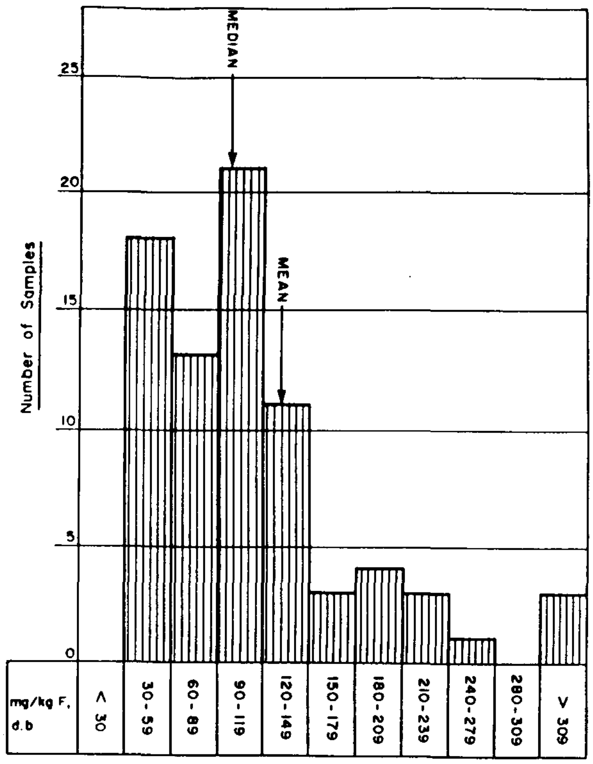

Figure 13
Fluoride concentrations in SA sewage sludges

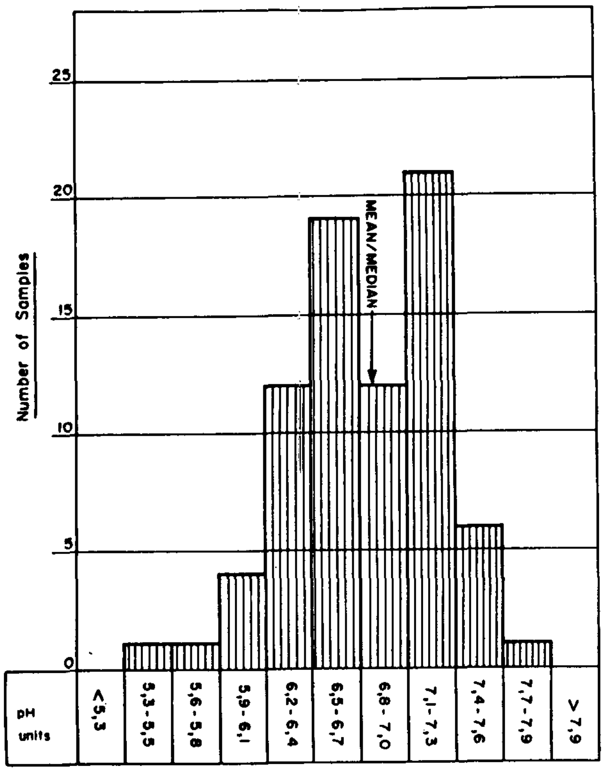

Figure 14
pH values of SA sewage sludges

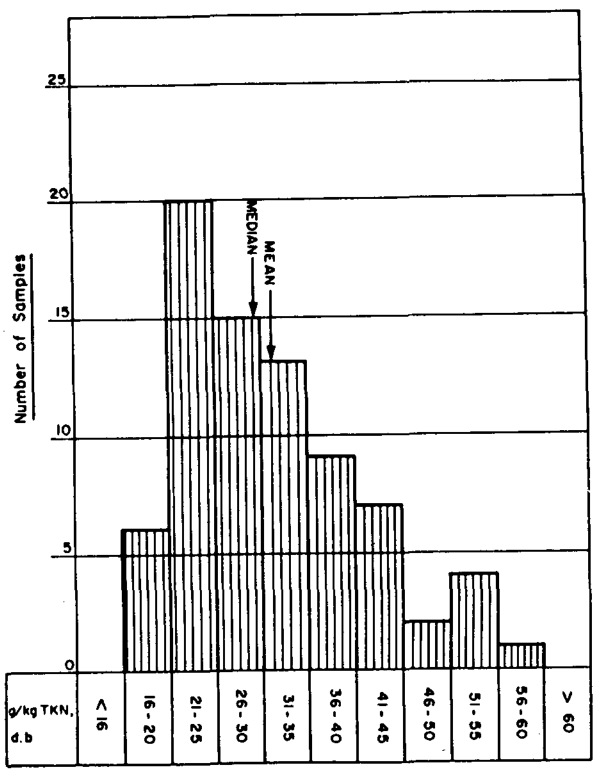

Figure 15
Total Kjeldahl nitrogen concentrations in SA sewage sludges

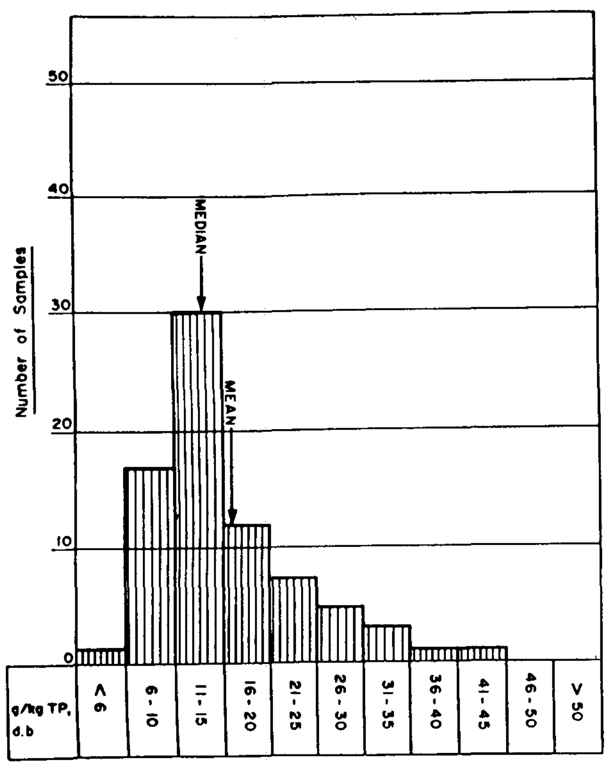

Figure 16
Total phosphorus concentrations in SA sewage sludges

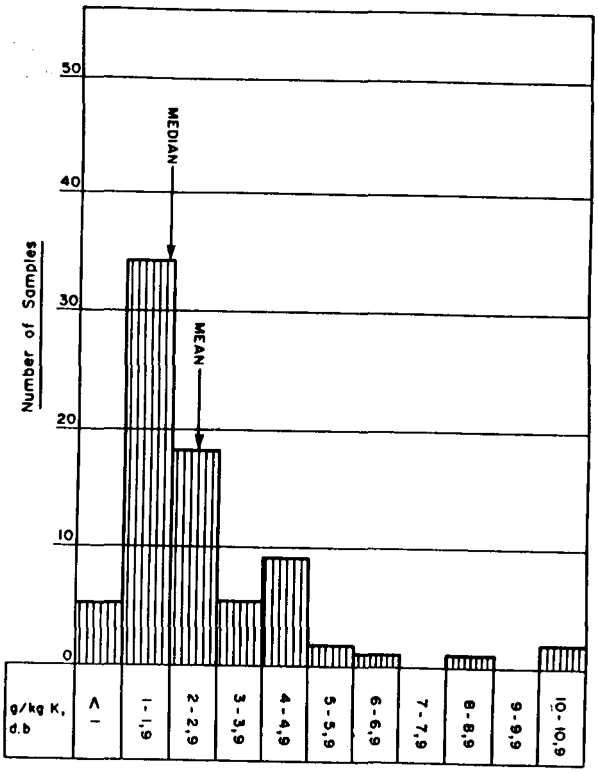

Figure 17
Potassium concentrations in SA sewage sludges

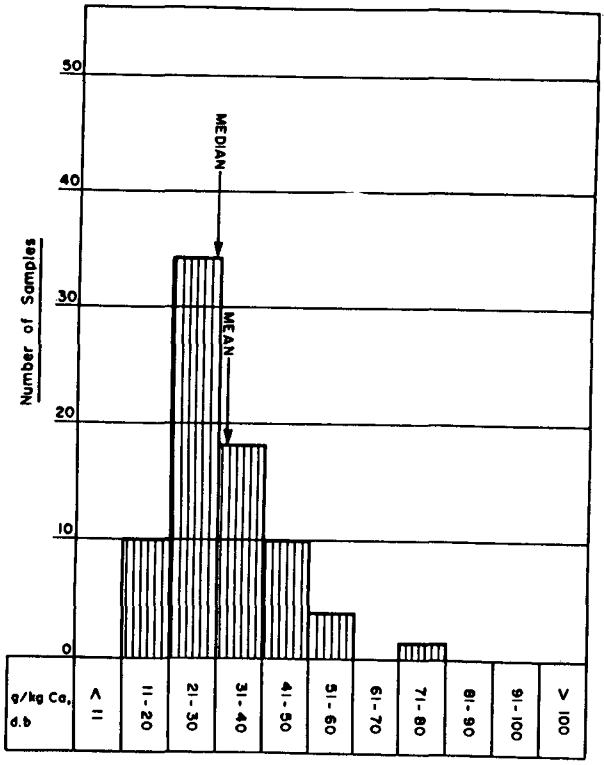

Figure 18
Calcium concentrations in SA sewage sludges

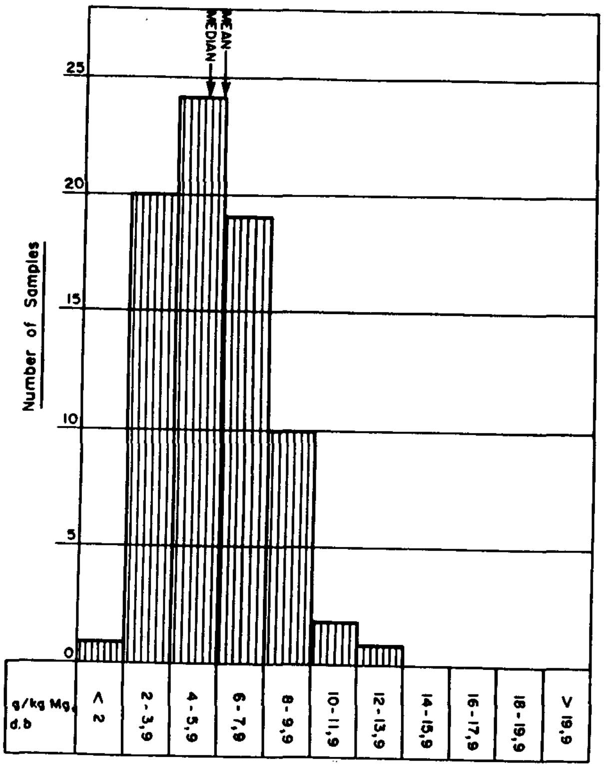

Figure 19
Magnesium concentrations in SA sewage sludges

Suggested guidelines for land application of sewage sludge

It is advisable that the application of disinfected sewage sludge to agricultural land be strictly controlled in order to minimise the risks associated with this practice. In formulating suggested inorganic chemical contaminant guidelines, cognisance was taken of the following:

- Existing overseas guidelines (Webber *et al.*, 1983; European Economic Community, 1986). These are shown in Table 6.
- Guidelines prepared by the National Institute for Water Research for the Department of National Health and Population Development (Table 7), based on a survey on the concentrations of heavy metals in the Cape Province, as well as on experimental lysimeters and plot studies. Limits were suggested for nine heavy metals in "Type D" sludge produced for unlimited use on land at a maximum application rate of 8 dry t/ha.a (Engelbrecht *et al.*, 1981; Kitshoff *et al.*, 1987; Vivier *et al.*, 1988).
- The results obtained from the analysis of sludges from seventy-seven South African sewage works as described in this paper.

Table 8 lists the proposed guidelines. Of the 77 sludges analysed, 45 were found to have met the suggested guidelines, 16 were over the suggested maximum permissible concentrations in the case of one contaminant only, while the remaining 16 exceeded the maximum concentrations for two or more contaminants.

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Natal : Amanzimtoti, Durban, Ladysmith, Newcastle, Pietermaritzburg, Port Shepstone.

Cape : Bellville, Cape Town, Cradock, De Aar, Despatch, East London, Graaff-Reinet, Grahamstown, Kimberley, King William's Town, Knysna, Kraaifontein, Milnerton, Oudtshoorn, Paarl, Parow, Port Elizabeth, Robertson, Stellenbosch, Strand, Uitenhage, Upington, Vredenburg/Saldanha, Walvis Bay, Worcester.

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TABLE 6
MAXIMUM PERMISSIBLE CONTAMINANT CONCENTRATIONS (mg/kg, DRY BASIS) IN SEWAGE SLUDGES FOR APPLICATION TO AGRICULTURAL LAND (OVERSEAS)

Country	Contaminant									
	Cd	Cr	Cu	Pb	Ni	Zn	Hg	As	Se	Mo
Belgium	10	500	500	300	100	2 000	10	10	25	-
Denmark	8	-	-	400	30	-	6	-	-	-
Finland	30	1 000	3 000	1 200	500	5 000	25	-	-	-
France	20	1 000	1 000	800	200	3 000	10	-	100	-
Germany	20	1 200	1 200	1 200	200	3 000	25	-	-	-
Netherlands	10	500	600	500	100	2 000	10	10	-	-
Norway	10	200	1 500	300	100	3 000	7	-	-	-
Sweden	15	1 000	3 000	300	500	10 000	8	-	-	-
Switzerland	30	1 000	1 000	1 000	200	3 000	10	-	-	20
Canada*	20	-	-	500	180	1 850	5	75	14	20
EEC-recommended	20	750	1 000	750	300	2 500	16	-	-	-
EEC-mandatory	40	-	1 500	1 000	400	3 000	-	-	-	-
Lowest recommended value	8	200	500	300	30	1 850	5	10	14	20
Highest recommended value	40	1 200	3 000	1 200	500	10 000	25	75	100	20
Mean of recommended values	19	795	1 430	690	235	3 485	12	32	25	20
Median recommended value	20	1 000	1 100	675	200	3 000	10	10	46	20

*Canada: Values apply to sludges and sludge-based commercial products containing <5% nitrogen

TABLE 7
MAXIMUM METAL CONTENT IN TYPE D SEWAGE SLUDGE

Metal	Maximum metal content (mg/kg dry sludge)
Cadmium	20
Cobalt	100
Chromium	2 750
Copper	750
Mercury	10
Molybdenum	25
Nickel	200
Lead	250
Zinc	2 750

TABLE 8
PROPOSED SOUTH AFRICAN GUIDELINES FOR MAXIMUM PERMISSIBLE CONCENTRATIONS OF INORGANIC CHEMICAL CONTAMINANTS IN SEWAGE SLUDGES FOR APPLICATION TO AGRICULTURAL LAND

Contaminant	Maximum permissible concentration (mg/kg, dry basis)
Cadmium	20
Chromium	1 500
Copper	1 000
Lead	750
Nickel	300
Zinc	3 000
Mercury	10
Arsenic	15
Selenium	15
Molybdenum	20
Boron	80
Fluoride	400
pH (pH units)	5,8 - 7,8

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