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ACID RECOVERY OF CADMIUM AND LEAD FROM IMMOBILIZED ACTIVATED SLUDGE USED TO BIOSORB METALS.

BY

1) CHANDRAKANTH HARTMAN

Thesis submitted to the Faculty of the Graduate School of the New Jersey Institute of Technology in partial fulfillment of the requirements for the degree of Master of Science.

Spring 1991

APPROVAL SHEET

Title of Thesis : ACID RECOVERY OF CADMIUM AND LEAD FROM IMMOBILIZED ACTIVATED SLUDGE USED TO BIOSORB METALS.

Name of the Candidate : Chandrakanth Hartman
M.S.in Environmental Science, 1991.

Advisor :

5/10/91
Date

Sam Sofer, Professor
Department of Chemical Engineering,
Chemistry, and Environmental Science,
and Research Chair in Biotechnology lab,
N.J.I.T.

Members :

5/10/91
Date

Richard Trattner, Professor
Associate Chair of Chemical Engineering,
Chemisty and Environmental Science, N.J.I.T.

May 10, 1991
Date

Henry Shaw, Professor
Department of Chemical Engineering,
Chemistry and Environmental Science, N.J.I.T.

VITA

Name : Chandrakanth Hartman

Permanent address : 198/321, 23rd cross,

6th block, Jayanagar,

Bangalore 560082

Karnatak State, INDIA.

Degree and Date to be conferred : M.S., 1991

Date of Birth :

Place of Birth :

Secondary education :

Collegiate institutions	Dates	Degree	Date
New Jersey Institute of Technology. Newark, N.J.	Aug 1989 to May 1991	M.S.,	May 1991
Gulburga University	May 1982 to May 1985	M.A.Sc.,	May 1985
Karnatak University	May 1978 to May 1981	B.Sc.,	May 1981

Major : Environmental Science.

ABSTRACT

Title of Thesis : Acid recovery of Cadmium and Lead from immobilized activated sludge used to biosorb metals.

Chandrakanth Hartman, M.S in Environmental Science. 1991

Thesis directed by : Sam S.Sofer, Professor,
Sponsored Chair in Biotechnology.

A study of the adsorption and subsequent desorption of heavy metals using various concentrations of hydrochloric acids has been performed.

To accomplish this task, the following areas were investigated : immobilization of the sludge, adsorption of heavy metals, various concentrations of acid used as stripping agents, examination of the relative fractions of heavy metals obtained using various stripping agents, and the actual concentration of the acid at a particular fraction.

It was observed that water helps in extraction of cadmium to a certain extent, but lead cannot be extracted with water. Also, water mixed with dilute hydrochloric acid is more efficient in extraction of metals as compared with direct addition of strong hydrochloric acid. It was also observed that nitric acid was not a very effective stripping agent as compared with hydrochloric acid.

PREFACE

This thesis presents a study done in work related to the biotechnology field.

Biotechnology is defined as "the application of scientific and engineering principles to the processing of organic and inorganic materials by biological agents to provide goods and services".[1]

Scientific and engineering principles cover a range of disciplines including biochemistry, microbiology, genetics, biochemical and chemical engineering and to some extent electrical engineering. The concept of "biological materials" refers to a wide range of biological catalysts such as enzymes, microorganisms, and animal and plant cells, while "goods and services" covers products of industries concerned with food, pharmaceuticals, biochemicals, metal recovery and biological waste treatment.

Heavy metals have been defined as "those with a specific gravity greater than 4 or 5, located from atomic numbers 22 to 34 and 40 to 52 on the periodic table (as well as the lanthanide and the actinide series), and having a specific biological response".[2]

The most common heavy metals include titanium, vanadium, chromium, manganese, iron, nickel, copper, zinc, arsenic, silver, cadmium, tin, mercury, and lead.

A discussion of bioaccumulation and toxicity of heavy metals requires a basic definition of terms. Toxicology is defined as "the science which studies the adverse effects of chemicals on living organisms and assesses the probability of their occurrence". With this definition in mind, the prime objective of any toxicology investigation is to provide the primary data base from which estimates of risk can be made for a given population of organisms.[2]

The area of wastewater treatment, gives scope to potentially exploit the intrinsic capability of microorganisms to "clean up" waters, especially since conventional physical and chemical means of removing soluble metal waste are generally very expensive when the contaminant concentrations are in the range of 10-100 ppm and below. Microorganisms have commonly been reported to be capable of sequestering heavy metals and concentrating them up to several thousand times over their concentration in the environment. With the increasing value and the growing scarcity of some metals, this inherent ability of the microorganisms has given prominence to the study of this field from the viewpoint of possible metal recovery. Some plants are also known to have the ability to concentrate specific metals, and their relative abundance has been used as indicators for "botanical metal prospecting". Also, some studies have been done to demonstrate the possible use of algae and simple plants as bioindicators of metal pollution in water systems.[3]

Many researchers have sought to exploit this behavior, and numerous investigations reporting a wealth of data of a qualitative nature have been published. Field and pilot plant studies on metal ion recovery/removal have been conducted and several patents have appeared. The uptake process, however, is very complex and is dependent on the metal ion and the biological system in question.[3]

The work study done here involves the use of immobilized bacteria to biosorb from water and to strip the heavy metals using different concentrations of acid solutions.

ACKNOWLEDGEMENTS

I wish to express sincere gratitude to my supervising professor, and Sponsored Chair in Biotechnology, at NJIT Dr. Sam S.Sofer for his guidance, friendship and moral support.

Special thanks are also due to Dr. Richard Trattner, graduate adviser and Dr. Henry Shaw, for being members on my masters committee.

I am also grateful to the State of New Jersey for funding through the Sponsored Chair in Biotechnology and to the Hazardous Substance Management Research Center in providing funds for further and continued research on this project.

A note of special thanks to Dr. Remy Gourdon, currently at the Laboratoire de Chimie Physique Appliquee et Environnement, France, who guided me during his appointment at the NJIT Biotechnology laboratory and has constantly kept in touch with me throughout this work study.

Appreciation is also expressed to Dr. Fayaz Lakhwala and Emilia Rus for their timely help.

Finally I would like to express my sincere appreciation to all the members of the NJIT Biotechnology Laboratory.

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CHAPTER I

INTRODUCTION

1.1 IMPACT OF HEAVY METALS ON ENVIRONMENT

The deterioration of environmental quality began when man first collected into villages and utilized fire. Since then this has existed as a serious problem. In the latter half of the twentieth century under the impacts of exponentially increasing population and industrialization the environmental contamination of air, water, soil, and food has become a threat to the existence of many plant and animal communities as well as the very survival of the human race.

The drinking water has been rendered detrimental due to the ubiquitous nature of heavy metals in some areas, and due to the traditional role of surface waters as receiving bodies for wastewater effluents. In addition, surface waters are increasingly being utilized for potable supplies as natural groundwater supplies are depleted and new sources become difficult to exploit. Thus, water is being increasingly reused both directly and indirectly at present. Indirect re-use of wastewater occurs when " water which is already used one or more times for domestic or industrial purposes is discharged to a fresh surface or an underground reservoir of water and is used again in its diluted form".[4]

The disposal of contaminated industrial wastes into streams, lakes and rivers causes an imbalance in the ecosystem of the water bodies. The industrial effluents like those of sugar industry, brewery, paper, textile and many others have mainly been a source of organic pollutants which get oxidised in the presence of microorganisms utilizing the dissolved oxygen of the water bodies. The effluents like those from electroplating and chloralkali industries and the drainage of mine works cause metal pollution when discharged into these water bodies thus creating a hazard to the aquatic life.[5]

The industrial and domestic activities which emit the heavy metals into the environment in many cases influence the activity of the microbial population. In the natural environment, the effects of heavy metals to a large extent depend on the chemical composition of the recipient body into which the metals are deposited.[6]

The contamination of the heavy metals is a very serious public health problem due to the toxicity of most of these pollutants even at low concentrations. The ability of many living organisms to accumulate them results in their concentration in the human food chain.[7]

The environmental impact of heavy metals discharged to the aquatic environment is controlled significantly by their speciation. The physicochemical form of heavy metals will determine their solubility, mobility and bioavailability. In the context of heavy metal pollution, wastewater treatment processes can be currently considered to be an integral component of the hydrological cycle. It has been calculated that several thousand tons of the heavy metals of major concern pass through wastewater treatment plants in the United Kingdom each year. The speciation of heavy metals in wastewater treatment processes is important for two major reasons. First, it can influence the formation of particulate forms of metals which are subsequently removed by sedimentation, thereby having important implications for maintenance of the quality of the receiving waters, which may in many locations constitute a source of abstraction for potable supply. Second, the physicochemical forms of the metals which are removed in the sludge produced will ultimately determine their environmental impact when the sludges are disposed to sea or the land.[8]

The selective accumulation of heavy metal ions by various microorganisms has also been examined. Uranyl, mercury and lead ions are readily accumulated by almost all the species of microorganisms.[9]

Probably the most important feature which distinguishes the heavy metals from other toxic pollutants is that they are not biodegradable and having entered the environment their potential toxicity is controlled to a great extent by biological and geochemical factors.[10]

1.2 SOURCES OF HEAVY METALS

Those elements which are known collectively as the "heavy metals" are a fairly ill defined group. Generally they include many of the transition series metals and some of the metals and metalloids in groups IIIB, IVB, VB and VIB of the periodic table. Although many of them are micro-nutrients, they are primarily of interest because of their toxic properties to all forms of life.[10]

The most important sources of lead include lead paints, some of which incorporate organolead oxides, lead water pipes, used motor oil, and gasolines containing tetraethyl lead or methylethyl lead additives. Much of the lead eventually makes its way into natural waters where it accumulates in sediments. Lead has also been found to accumulate in sewage sludges (up to 7627 ppm) of American cities along with many other heavy metals, the distribution of metals being characteristic to each city. Recent evidence indicates that lead may also be methylated by bacteria.[11]

Cadmium is a relatively rare element and is present in the natural environment at relatively low levels. The average concentration is 0.06 ppm in soils, 0.08 ppm in fresh water and 0.1 ppb in sea water. The plants have a tendency to adsorb cadmium, the highest levels being reported in Japan near the vicinity of mines and smelters. Persons living in these areas exhibited "itai-itai" disease, characterized by osteomalacia and severe pain in the skeletal mass. In the United States, maximum concentration limits from cadmium in potable water are set at 0.01 ppm. Sources of pollution include by-products of metal processing mine wastes, electroplating wastes, fertilizers, pesticides, and nuclear reactors as neutron control rods which leads to increasing levels of the Cd metal in the environment. The cadmium content of rivers, lakes and sludges has been reported to be increasing with time. Although cadmium, has been shown to accumulate in plants, marine plankton and invertebrates, no bioaccumulation has been reported in fish. The nature and extent of cadmium interaction with such biota appear highly dependent on the molecular species of cadmium involved.[5,11]

Cadmium is also produced during smelting, electroplating and pigment manufacture, while other metals are used in, and generated as radioisotopic products of nuclear fission processes in the nuclear power, defense, and fuel reproducing industries. Heavy metals are toxic to living organisms both per se and by the radiotoxicity of isotopes that may be ingested.[12]

Hence studies on the accumulation and biotransformation of cadmium by bacteria are of special interest, as the final chemical form of cadmium is likely to dictate its relative toxicity.[13]

1.3 TYPES OF TREATMENT DONE IN THE FIELD

Wastewater treatment requires the knowledge of the ways in which heavy metals ions and activated sludge flocs interact. In the aeration tank, adsorption of metals by the activated sludge affords protection for the receiving watercourse. When sludge disposal is being considered, the strength of the metal binding affects the readiness with which metals are ultimately dispersed into the environment. In addition to these more obvious roles, metal ions can be expected to form an integral, possibly significant, part of the floc structure. As such, the adsorption of polyvalent cations will affect the settlement of sludge. Conversely, because the nature of the sludge surface varies with the settlement characteristics, the ability of the sludge to bind metals can be expected to alter as the settlement properties change.[14]

Several chemical methods for solublization of heavy metals from sewage sludge have been suggested. They include acidification, chemical chelation, liquid ion exchange, chlorination, and oxidative acid hydrolysis. However, factors such as cost, operational difficulties, and in some cases unacceptable metal solubilization efficiencies have made practical application of these chemical methods unattractive.[15]

Although a precipitation stratagem may result in removal of soluble cadmium, it does not necessarily extract the metal in a manner that allows for easy subsequent isolation from the solution, as would be the case with adsorption onto particle (cell) surfaces.[16]

Biological treatments provide promising alternative techniques. Scientific papers and patents in the area are numerous. However, most of the studies have involved single microbial species, which may be a limitation to field applications. The use of activated sludge appears as a more attractive solution because it is available at very low costs and may be readily used as a biosorbent without additional facilities which would be required to grow pure species. Another advantage of activated sludge is its capability to biodegrade a large variety of organic contaminants which may be found along with heavy metals in many liquid wastes.[7]

The use of sewage sludge on land has been an appealing sludge disposal method in U.S in the last decade, especially in the areas where available land is abundant compared to the disposal needs because much of the sludge applied to the crop land allows efficient use of sludge nutrients and also the sludge acts as a soil conditioner and as a fertilizer. However, the major limitation with land application of sewage sludge has been the high heavy metal concentration in the sludge. The reduction of heavy metals content of sewage sludge could significantly increase the amount of sludge suitable for land application.[15,17]

An alternative microbial strategy to decrease toxic threat is transformation of the metal into non toxic or non assimilable form. This may be achieved intra- or extracellularly by processes such as oxidation, reduction or methylation.[16]

1.4 FACTORS AFFECTING METAL REMOVAL IN ACTIVATED SLUDGE

Metal solubilities are greatly affected by other factors, such as acidity therefore in practice it is only possible to make a crude estimation of the expected solubility of any one metal. Activated sludge treatment is a dynamic process, the influent metal concentrations are continually changing hence equilibria between the phases of the metals are continually shifting.[18]

Many different factors have been shown to affect the removal of metals in the activated sludge process. They may be divided into plant operating parameters, physical or chemical factors, and biological factors. Operating parameters which have been shown to affect metal removal are sludge volume index (SVI), sludge age,suspended solids removal, mixed liquor suspended solids concentration (MLSS), dissolved oxygen concentration, and settling time. Physical and chemical factors affecting metal removal are pH, temperature, metal-ion concentration,metal solubility, metal valency, concentration of complexing agents, and particle size. The principal biological factor is the concentration of extracellular polymers. However, other microbial products including metabolic intermediates and materials released as the result of lysis cannot be ignored. Bacterial extracellular polymers attached to cells and any material associated with them will bind soluble metal to the cell.[19,20]

The heavy metals are concentrated in the sludges during sewage treatment. These metals are strongly combined with the sludge solids and must be solubilized before they can be removed. Following metals solubilisation, the sludge has to be dewatered and the metal laden liquor collected for metals removal. Finally, the decontaminated sludge solids and liquor can be recombined and neutralized for land application.[15]

There has been an increasing concern about the amount and quality of sewage sludge that has to be disposed in the recent years. As of today, every person in any way connected with mechanical-biological sewage treatment produces on an average 0.5 m^3 yr^{-1} of stabilized sewage sludge containing, 0.5 g of Cd, 0.1 g of Hg, and 0.03 g of polychlorobiphenyl (PCB). The traditional utilization of sewage sludge on farmland has become open to question because of the increase in the metal content of the soil and recently, also because of the presence of toxic organic constituents in high concentrations, like alkylated phenols. Thermal treatment is considered an alternative to the agricultural use of sewage sludge and the disposal of resultant slag and ashes in sanitary landfills. The main problem is the fate of inorganic and organic sludge constituents during the thermal treatment and final disposal. Several studies deal with the release of heavy metals and the destruction of organic materials during the incineration of sludge.[21]

The primary sedimentation is used to remove particulate and precipitated material, which generally accounts for up to 40 % of the initial metal load. Forms of metal entering the biological treatment stage are predominantly soluble. Biological removal of soluble metals is critical because their overall retention depends on absorption by the activated sludge biomass.[20]

The secondary biological treatment favors the metal removal efficiency which is comparable with or slightly better than those in primary sedimentation, but the wide variation in reported removal efficiencies indicates the complexity of the process.

While industrial pretreatment remains the most effective means of reducing metals in sludge, several studies have investigated the feasibility of removing metals from sludge. These have all involved acid extraction and most have used activated sludges. The emphasis here, however, is on the acid-extraction process itself and the factors that affect Cd and Pb removal[17].

1.5 ADVANTAGES OF ACID EXTRACTION PROCESS

The acid extraction process is a conventional method of stripping the heavy metals from the contaminated sludge. This method is selected because dilute acids are easier to handle and the metals have a tendency to be washed easily by the acid than other chemicals such as surfactants. This method is less time consuming as compared to surfactant washing and the contact time between the acid and the heavy metal is not very important as compared to surfactants where significant contact time between the metal ion and the surfactant is neccesary.

1.6 ACCEPTANCE LIMITS

Cd, Pb and other heavy metals are the metals of most concern. The average concentration of lead in the earth's lithosphere has been reported to be 16 ppm, with a range of 2-200 pm. Average values of lead in soils, freshwater, and seawater have been reported to be 10, 0.005, and 0.00003 ppm respectively. The maximum total lead concentration permitted in United states potable waters is 50 ppb. Much higher concentrations are found in soils near lead mining operations and highways. Lead is absorbed in plant and animal tissues where it accumulates to a greater extent than mercury or cadmium. This is very much true of plants growing in heavily contaminated soils near roadways[7].

By whatever method water quality control standards are used, the movement to reduce and ultimately eradicate aquatic pollution is likely to require increased restrictions on the discharge of metal-laden, and other effluents. To comply with the standards, industrial effluents could be diverted from direct discharge to sewers, which could cause treatment plant performance to deteriorate but which would improve the condition of receiving water. Thus, as standards are raised, wastewater treatment will contribute significantly to the maintenance of surface water quality.[20]

1.7 THE THRUST OF THIS WORK

The work done here is part of a larger study involving the use of immobilized bacteria to biosorb the heavy metal content of the wastewater sludges and to subsequently strip the heavy metals using different concentrations of acid solutions. The exponential gradient gives sharp concentration peaks for the heavy metals extracted, and this feature is attractive for recovering the metals. It is also possible that further work will allow selective removal of heavy metals from each other in the same batch. This would, with some refinement, yield an attractive and re-usable system for treatment of heavy metals in water.

CHAPTER II

OBJECTIVES

The primary objective of this study is to evaluate the technical feasibility of recovering metals by acid washing of the biosorbent.

The first step has been to develop a procedure for a quantitative evaluation of the recovered metals from the contaminated beads. This was explored by washing the contaminated beads with water, and varying concentrations of hydrochloric acid and nitric acid. The estimation of the metals removal was done by an analysis on the atomic absorption spectrophotometer.

The secondary objective is to determine the technical feasibility on a laboratory scale of the entire process for selective removal of heavy metals from industrial wastes.

Accomplishment of the primary objective involves the following steps :

1. Investigation of the removal of heavy metals like cadmium and lead using hydrochloric acid at various concentrations. (0.1 N, 1.0 N, and 5.0 N HCl).

2. A comparative study using different concentrations of nitric acid and hydrochloric acid. This study highlights the feasibility of extracting heavy metals using a particular acid solution at a particular concentration.

3. A study to observe the concentrations of the acid solutions which give the maximum removal.

Accomplishment of the secondary objective involves the extraction of heavy metal mixtures containing both Cd and Pb.

CHAPTER III

LITERATURE SURVEY

Many persistent anthropogenic pollutants are present in the raw industrial and domestic waste waters. One group, the heavy metals, is particularly hazardous. Therefore, these materials must be removed from the effluents before they potentially cause harm to both the ecosystem of the receiving watercourse and the public health. Adequate dilution capacity of receiving waters is important to minimize the adverse effects of effluents, especially in areas where water reuse is practiced. In other areas with extensive water reuse, almost 90 % of surface flow could be from wastewater effluents. Because of the nature of wastewater teatment processes, accumulation of mineral content is inherent in water reuse, thus, considerable environmental concern has focused on non-biodegradable and toxic heavy metals.[20]

Lead normally enters the environment by potential sources such as lead paints, lead water pipes, motor oil, and gasoline containing tetraethyl lead. It also makes its way into the natural waters and accumulates as sediments.

Cadmium is a relatively rare element with no known biological function. It is normally found in the natural environment at lower concentrations. Various anthropogenic sources, such as byproducts from coal combustion, mine wastes, fertilizers, electroplating processes, and iron and steel industries are the potential sources of cadmium entering the environment.

High concentrations of heavy metals are toxic to most microorganisms and often cause serious upsets in a biological waste treatment system. However, when the metal concentration is not too high, and a proper acclimation process is allowed, a biological system may be used to remove a certain amount of metal without itself being adversely affected.[22]

When sludge disposal is being considered, the strength of the metal binding affects the readiness with which metals are ultimately dispersed into the environment. The distinctions between active and passive metal uptake were made by Lawson *et al*.,[19] using activated sludge deactivated with formaldehyde.

The removal of soluble cadmium by precipitation was studied by J.A.Scott *et al*. [16]. Precipitation does not necessarily extract the metals in a manner that allows for easy subsequent isolation from the solution, as would be the case with adsorption onto cell surfaces.

The optimum pH required for the maximum biosorption of Cd was studied by Gourdon, *et al*.[7], of our laboratory, where they indicate that pH is an important factor in the study of Cd uptake by microorganisms. Using similar conditions the study was also done in extraction of lead [7].

While many effective treatment methods of reducing metals in sludge are present, only few studies have investigated the feasibility of removing metals from the sludge. Terry Logan and Robert Feltz [17] demonstrated that Cd could be extracted using acid effectively. Acid extraction by exponential gradient was used for the extraction of Pb and Cd in this study.

CHAPTER IV

MATERIALS AND EXPERIMENTAL METHODS

4.1 SLUDGE FOR CADMIUM AND LEAD EXTRACTION

Two liters of sludge were collected from the Parisipany water treatment plant, NJ in plastic buckets. The sludge was aerated with atmospheric air forced through a sparger which was used to create smaller bubbles to increase the surface area of the liquid-air interface and improve gas transfer. After 24 hours the aeration was stopped and the sludge was sieved through 500 um sieve to remove inert gross particles and the sludge was frozen and stored at -20° C to prevent changes in biomass composition. Later the sludge was centrifuged to prepare pellets. These pellets were later used to prepare beads using calcium alginate.

The dry mass of the biomass was calculated and about 200 mg dry mass of biobeads were used for the experiments.

4.2 IMMOBILIZATION

The entrapment of microorganisms in calcium alginate gel was carried out as follows. Distilled water and concentrated pellets were taken in a ratio of 5 : 2 by weight along with sodium chloride (0.5 % w/w) in a blender. Sodium alginate (1.5 % w/w) was added slowly to the mixture, with continuous stirring to obtain a homogeneous cell suspension. With the help of a syringe pump, the homogeneous cell suspension was then extruded as discrete droplets in a slowly stirred solution of 0.1 M calcium chloride. On contact with calcium chloride, the droplets hardened to form beads about 3-3.3 mm in diameter. Here calcium chloride acted as a cross-linking agent. The beads were then cured in calcium chloride for 24 hours at 4°C before use.

4.3 CADMIUM EXTRACTION

About 70 gms of untreated biobeads were taken in a flask and washed with water and about 550 mg Cd was added to the flask with 180 ml of 0.5 % NaCl to prevent the bursting of the bacterial cells. The pH 6.0 was adjusted to allow maximum biosorption of cadmium in the beads over a period of 24 hours in a shaker. Later the flask was removed from the shaker and the solution was analyzed for cadmium. The difference in the concentration of Cd before and after the biosorbtion gave the actual mass of Cd absorbed in the beads. The total mass of Cd in the beads was found to be 261.2 mg.

The amount of cadmium taken for the extraction experiment was about 14.541 mg which corresponds to about 3.897 g wet beads (200 mg dry biomass) which were packed in a 1 cmID column. The bed height was typically 6 cm. The Cd - laden biomass was washed with about 100 ml water. And the column was flushed with an exponentially increasing concentration gradient of HCl as a stripping solution. Every 25 ml of the fraction was collected in a beaker and analyzed for Cd and the concentration of the acid.

4.4 LEAD EXTRACTION

The same type of conditions mentioned above for the extraction of cadmium were used and about 550 mg of lead was added to about 70 gms of beads and the pH was maintained at about 5.0. After 24 hours of biosorption the actual amount of lead contained in the 70 gms of beads was about 539.98 mg.

The mass of lead taken for the extraction experiment was 26.84 mg which corresponds to about 3.48 g wet beads (200 mg dry mass).

4.5 ANALYTICAL METHODS

Both cadmium and lead concentration were determined by atomic absorption on a Perkin-Elmer spectrophotometer. The operating conditions were as recommended in the Perkin-Elmer instruction manual, and with these standard conditions the accuracy was estimated at +/- 2 % .

CHAPTER V

RESULTS AND DISCUSSION

5.1 STRIPPING OF LEAD WITH 0.1 N HCl

About 100 % of the lead was extracted using the above technique and the runs were done in duplicate maintaining the same conditions mentioned above. It can be clearly observed in Table 1 that at a concentration of about 0.04 N HCl about 11.77 mg of lead was extracted, which amounts to about 43.85 % of lead extraction. Also we observe from the same table that about 89.8 % of lead was off between fraction 8-10. See Fig 1 and 2.

Table 1 : Extraction of Lead with 0.1 N H HCl

Fraction #	Volume of elution	mg of lead	Concn. HCl in N
1	25 ml	0.05	0.0
2	25 ml	0.043	0.0
3	25 ml	0.012	0.0
4	25 ml	0.012	0.0
5	25 ml	0.037	0.001
6	25 ml	0.0125	0.001
7	25 ml	0.762	0.002
8	25 ml	11.77	0.04
9	25 ml	9.83	0.085
10	25 ml	2.23	0.093
11	25 ml	0.66	0.094
12	25 ml	0.418	0.096
13	25 ml	0.268	0.097
14	25 ml	0.225	0.1
15	25 ml	0.206	0.1
Total	375 ml	26.535	

initial Pb taken = 26.84 mg; total Pb extracted = 98.86 %

5.2 STRIPPING OF LEAD WITH 1.0 N HCl

Table 2 indicates that 0.03 N HCl gives the maximum extraction of 12.5 mg of lead which corresponds to about 46.57 % of lead extraction. This indicates that a slight acidic concentration enhances the efficiency of the metal recovery. See Figures 3 and 4.

Table 2 : Extraction of lead with 1.0 N HCl

Fraction #	Volume of elution	mg. Lead.	Concn. of HCl in N
1	25 ml	0.0	0.0
2	25 ml	0.0	0.0
3	25 ml	0.0	0.0
4	25 ml	0.0	0.0
5	25 ml	12.50	0.03
6	25 ml	5.68	0.08
7	25 ml	2.05	0.21
8	25 ml	0.71	0.31
9	25 ml	0.12	0.39
10	25 ml	0.09	0.47
11	25 ml	0.02	0.55
12	25 ml	0.01	0.65
13	25 ml	0.00	0.74
14	25 ml	0.00	0.87
Total	350 ml	21.19 mg.	

initial Pb taken = 26.84 mg; total Pb extracted = 78.95 %

5.3 STRIPPING WITH 5.0 N HCl FOR LEAD

Table 3 indicates that when the concentration of HCl is about 0.62 N about 8.5 mg of lead was extracted corresponding to 31.6 % of the mass of lead initially taken. This indicates that higher concentrations of acid may not necessarily contribute to added extraction. It also indicates that mild acid might provide better extraction at a particular fraction. See Figures 5 and 6.

Table 3 : Extraction of lead with 5.0 N HCl

Fraction #	Volume of elution	mg of lead.	Concn of HCl in N
1	25 ml	0.00	0.0
2	25 ml	0.03	0.0
3	25 ml	0.043	0.0
4	25 ml	0.075	0.0
5	25 ml	8.493	0.618
6	25 ml	3.881	1.237
7	25 ml	2.468	1.904
8	25 ml	1.937	2.380
9	25 ml	1.706	2.761
10	25 ml	1.568	3.237
11	25 ml	1.50	3.856
12	25 ml	1.393	4.046
13	25 ml	1.081	4.189
14	25 ml	0.881	4.330
Total	350 ml	25.057 mg	

initial Pb taken = 26.84 mg; total Pb extracted = 93.35 %

5.4 STRIPPING OF LEAD WITH 0.1 N HNO3

Table 4 indicates that when the strength of nitric acid is about 0.04 N the maximum extraction in a particular fraction is 9.12 mg which amounts to about 33.97 % of the initial mass of lead taken. See Figures 7 and 8.

When compared with 0.04 N HCl, we observe from Table 1 that the extraction of lead was about 43.85 % , which was almost 10 % greater. This indicates that at 0.04 N HCl is more efficient as compared to 0.04 N HNO_3.

Table 4 : Extraction of lead with 0.1 N HN03

Fraction #	Vol. of elution	mg of lead	Concn.of HN03 in N
1	25 ml	0.0	0.0
2	25 ml	0.0	0.0
3	25 ml	0.0	0.0
4	25 ml	0.0	0.0
5	25 ml	0.250	0.002
6	25 ml	0.500	0.015
7	25 ml	0.875	0.026
8	25 ml	9.125	0.040
9	25 ml	7.650	0.053
10	25 ml	3.000	0.069
11	25 ml	1.168	0.082
12	25 ml	0.500	0.088
13	25 ml	0.125	0.094
14	25 ml	0.100	0.100
Total	350 ml	23.293 mg	

initial Pb taken = 26.84 mg; total Pb extracted = 86.78 %

5.5 STRIPPING Of LEAD WITH 1.0 N HNO3

Table 5 indicates that 0.02 N nitric acid has a maximum extraction of 10.26 mg which amounts to about 38.22% of the initial amount of lead taken. See Figures 9 and 10.

Comparing the above results with 0.03 hydrochloric acid, in Table 2, we observe that about 46.57 % of lead was extracted. Nearly 8.35 % of lead was extracted in excess by using 0.03 N hydrochloric acid as compared with 0.02 N nitric acid. This indicates that 0.03 N HCl is more efficient as compared with 0.02 N HNO_3.

Table 5 : Extraction of lead with 1.0 N HNO3

Fraction #	Vol.of elution	mg of lead	Concn. of HNO3 in N
1	25 ml	0.037	0.0
2	25 ml	0.037	0.0
3	25 ml	0.037	0.0
4	25 ml	0.062	0.0
5	25 ml	10.262	0.02
6	25 ml	7.662	0.12
7	25 ml	2.612	0.176
8	25 ml	1.412	0.274
9	25 ml	0.900	0.353
10	25 ml	0.612	0.470
11	25 ml	0.425	0.588
12	25 ml	0.325	0.700
13	25 ml	0.275	0.830
14	25 ml	0.162	0.890
Total	350 ml	24.822 mg	

initial Pb taken = 26.84 mg; total Pb extracted = 92.48 %

5.6 STRIPPING OF LEAD WITH 5.0 N HNO3

Table 6 shows that when the strength of nitric acid is about 0.48 N the maximum amount of lead extracted is about 11.54 mg which corresponds to about 43 % of the extraction. See Figures 11 and 12.

By comparing the above results obtained with 0.6 N hydrochloric acid in Table 3, we observe that about 31.6% of lead was extracted in this particular concentration. And 0.48 N nitric acid seems to be more efficient since the lead extracted was more than 12 % .

Table 6 : Extraction of lead with 5.0 N HNO3

Fraction #	Vol.of elution	mg of lead	Conc.of HNO3 in N
1	25 ml	0.05	0.0
2	25 ml	0.05	0.0
3	25 ml	0.05	0.0
4	25 ml	0.112	0.0
5	25 ml	11.537	0.482
6	25 ml	8.250	1.010
7	25 ml	3.800	1.689
8	25 ml	0.681	2.316
9	25 ml	0.537	2.847
10	25 ml	0.262	3.185
11	25 ml	0.200	3.571
12	25 ml	0.062	4.053
13	25 ml	0.025	4.488
14	25 ml	0.012	4.632
Total	350 ml	25.628 mg	

initial Pb taken = 26.84 mg; total Pb extracted = 95.48 %

5.7 STRIPPING OF CADMIUM WITH 0.1 N HCl

By referring Table 7 we observe that when the strength of HCl is about 0.01 N, then the maximum extraction of cadmium in that particular fraction is 2.16 mg which corresponds to 14.86 % extraction of the initial mass of cadmium taken. See Figures 13 and 14.

Table 7 : Extraction of Cadmium with 0.1 N HCl

Fraction #	Vol of elution	mg. Cadmium	Conc.of HCl in N
1	25 ml	1.330	0.0
2	25 ml	0.417	0.0
3	25 ml	0.160	0.0
4	25 ml	0.102	0.0
5	25 ml	0.930	0.003
6	25 ml	2.162	0.010
7	25 ml	1.895	0.020
8	25 ml	1.385	0.028
9	25 ml	1.000	0.038
10	25 ml	0.697	0.049
11	25 ml	0.492	0.060
12	25 ml	0.302	0.072
13	25 ml	0.230	0.079
14	25 ml	0.190	0.086
Total	350 ml	11.294 mg	

Initial Cd taken = 14.541 mg; total Cd extracted = 77.67 %

5.8 STRIPPING OF CADMIUM WITH 1.0 N HCl

Here, Table 8 shows that at 0.04 N HCl the maximum extraction would be 4.1 mg of cadmium, which indicates that about 28.2 % of cadmium was extracted in that particular fraction. See Figures 15 and 16.

Table 8 : Extraction of cadmium with 1.0 N HCl

Fraction #	Vol of elution	mg. Cadmium.	Concn of HCl in N
1	25 ml	1.355	0.0
2	25 ml	0.550	0.0
3	25 ml	0.275	0.0
4	25 ml	0.127	0.0
5	25 ml	4.100	0.039
6	25 ml	3.395	0.196
7	25 ml	1.620	0.29
8	25 ml	0.807	0.39
9	25 ml	0.402	0.48
10	25 ml	0.335	0.5
11	25 ml	0.182	0.588
12	25 ml	0.130	0.637
13	25 ml	0.060	0.70
14	25 ml	0.025	0.70
Total	350 ml	13.364 mg	

Initial Cd taken = 14.541 mg; total Cd extracted = 91.90 %

5.9 STRIPPING OF CADMIMUM WITH 5.0 N HCl

It can be observed from Table 9 that when the strength of the HCl is about 0.23 N, the maximum extraction in that fraction was about 4.16 mg which corresponds to about 28.6 % extraction. See Figures 17 and 18.

Table 9 : Extraction of cadmium with 5.0 N HCl

Fraction #	Vol of elution	mg. Cadmium.	Conc.of HCl in N
1	25 ml	1.107	0.0
2	25 ml	0.475	0.0
3	25 ml	0.307	0.0
4	25 ml	0.205	0.0
5	25 ml	4.160	0.23
6	25 ml	3.085	0.66
7	25 ml	1.470	1.095
8	25 ml	0.900	1.571
9	25 ml	0.640	2.09
10	25 ml	0.450	2.523
11	25 ml	0.300	2.904
12	25 ml	0.227	3.237
13	25 ml	0.170	3.475
14	25 ml	0.145	3.665
Total	350 ml	13.641 mg	

Initial Cd taken = 14.541 mg; total Cd extracted = 93.81 %

5.10 STRIPPING OF CADMIUM WITH 0.1 N HNO3

Table 10 indicates that when the strength of nitric acid is about 8 x 10^{-3} N, the maximum extraction would be 2.27 mg which corresponds to 15.61 % of extraction.

When compared with Table 7, we observe that when the concentration of hydrochloric acid is about 0.01 N, the amount of cadmium extraction was about 14.86 %, which indicates that nitric acid is more efficient than hydrochloric acid when the concentration is approximately 0.008 N. See Figures 19 and 20.

Table 10 : Extraction of cadmium with 0.1N HNO3

Fraction #	Vol.of elution	mg Cadmium	Conc.of HNO3 in N
1	25 ml	1.22	0.0
2	25 ml	0.53	0.0
3	25 ml	0.20	0.0
4	25 ml	0.115	0.0
5	25 ml	0.995	0.002
6	25 ml	2.275	0.008
7	25 ml	1.565	0.020
8	25 ml	1.070	0.030
9	25 ml	1.000	0.041
10	25 ml	0.650	0.053
11	25 ml	0.460	0.064
12	25 ml	0.352	0.074
13	25 ml	0.200	0.081
14	25 ml	0.150	0.087
Total	350 ml	10.782 mg	

Initial Cd taken = 14.541 mg; total Cd extracted = 74.15 %

5.11 STRIPPING OF CADMIUM WITH 1.0 N HNO3

Table 11 indicates that at 0.17 N HNO3 the maximum extraction would be 3.067 mg of cadmium which in turn corresponds to about 21 % of cadmium extraction.

By comparing the above results with Table 8, we observe that when the concentration of hydrochloric acid was about 0.19 N, the amount of cadmium extraction was about 23.34 % which indicates, that hydrochloric acid is slightly better than nitric acid when the concentration is nearly equal to 0.17 N. See Figures 21 and 22.

Table 11 : Extraction of cadmium with 1.0 N HNO3.

Fraction #	Vol.of elution	mg Cadmium	Conc.of HNO3 in N
1	25 ml	1.66	0.0
2	25 ml	0.352	0.0
3	25 ml	0.152	0.0
4	25 ml	0.102	0.0
5	25 ml	2.262	0.02
6	25 ml	3.067	0.176
7	25 ml	1.522	0.333
8	25 ml	0.805	0.441
9	25 ml	0.442	0.539
10	25 ml	0.355	0.617
11	25 ml	0.262	0.705
12	25 ml	0.235	0.803
13	25 ml	0.135	0.803
Total	325 ml	11.351 mg	

Initial Cd taken = 14.541 mg; total Cd extracted = 78.06 %

5.12 STRIPPING OF CADMIUM WITH 5.0 N HNO3

It can be observed from Table 12, that when the strength of nitric acid is about 0.43 N , the maximum extraction of cadmium was 3.8 mg which is equal to 26.13 % of cadmium being extracted. See Figures 23 and 24.

When compared with Table 9, it is found that when the concentration of hydrochloric acid is about 0.66 N , about 21.21 % of cadmium was extracted. This indicates that nitric acid is more efficient than hydrochloric acid at when the concentration of acid is nearly 0.4 normal.

Table 12 : Extraction of cadmium with 5.0 N HNO3

Fraction #	Vol.of elution	mg Cadmium	Concn.of HNO3 in N
1	25 ml	1.815	0.0
2	25 ml	0.565	0.0
3	25 ml	0.102	0.0
4	25 ml	0.120	0.0
5	25 ml	3.815	0.434
6	25 ml	2.035	0.868
7	25 ml	1.000	1.399
8	25 ml	0.655	2.123
9	25 ml	0.425	2.700
10	25 ml	0.227	3.088
11	25 ml	0.220	3.474
12	25 ml	0.150	3.764
13	25 ml	0.140	4.150
14	25 ml	0.107	4.488
Total	350 ml	11.376 mg	

Initial Cd taken = 14.541 mg; total Cd extracted = 78.23 %

5.13 Mixture runs using both cadmium and lead together in the column

Washing the contaminated beads with water alone extracted some amount of cadmium initially. A series of runs was conducted using a batch of beads contaminated with both cadmium and lead together column at varying concentrations of hydrochloric acid. It was interestingly observed that almost 86-96 % of cadmium was stripped without any resistance from the contaminated beads immaterial of the concentrations of the acid used.

Extraction of lead was totally dependent on the concentration of the acid as can be observed in Tables 13, 14, 15 and 16 and the Figures 27 to 34. Lesser the concentration of the acid, lesser was the amount of lead stripped. The maximum lead that could be stripped using 0.005 N HCl, 0.01 N HCl, 0.05 N HCl and 0.1 N HCl was between 28-68 % . Tables 17 and 18 give a fair idea of the cumulative percent recovery of both lead and cadmium.

CHAPTER VI

CONCLUSIONS AND RECOMMENDATIONS

6.1.1 LEAD

It was first observed that water alone is insufficient to extract lead from the contaminated beads. It is clearly seen that in case of lead extraction a very dilute strength of HCl between 0.03 N and 0.04 N HCl gives maximum extraction between 43-46 % as observed in Tables 1 and 2, but in contrast a higher strength of the acid gives lesser extraction of lead. This can be clearly observed in Table 3, wherein the strength of the acid is about 0.62 N, and the maximum extraction was about 31.6 % .

By observing Tables 4, 5, and 6 as summarized in Figure 25 we can assume that nitric acid is not as efficient as HCl, and it contributes less in the extraction of lead.

6.1.2 CADMIUM

It was observed that water alone mixed with dilute hydrochloric acid was sufficient to extract almost all the cadmium present in the beads. The comparison with nitric acid can be done by referring Figure 26.

In fact, water contributed to some cadmium removal and later dilute hydrochloric acid enhanced the extraction of cadmium nearly to about 93 %. This can be observed by referring Table 9.

It can be observed from Table 12 that nitric acid is not as efficient as hydrochloric acid, and the maximum extraction of cadmium was about 78 % .

6.1.3 MIXTURE RUNS OF CADMIUM AND LEAD

By referring Figure 33, we observe that higher acidity peaks are sharper and the peaks overlap making the selective desorption difficult.

Lower concentration of the acid peaks are broad as seen in Figure 27. This indicates that it is possible to device the process for selective metal recovery using lower concentrations of acid. This would, with some refinement, yield an attractive and re-usable system for treatment of heavy metals.

6.2 INDUSTRIAL RELEVANCE

The information provided in the thesis might provide the basis for an innovative and cost-effective technology for the decontamination of a synthetic waste stream containing metals. This later can be scaled for field use pending engineering and economic evaluations. In addition, the biological process utilized in this system will provide for the removal and subsequent recovery of metals from the wastewater.

This project is of special interest, if it be an alternative to metal hydroxide precipitation for several reasons which include :

1. The materials generated by the biosorption process are "clean" water and recyclable metals versus treated water and a precipitated sludge requiring additional treatment and/or disposal.

2. This process utilizes a waste material (activated sludge) which can generally be obtained at low cost to treat another waste, whereas precipitation method requires the use of relatively costly non-renewable feed stocks such as sodium hydroxide and alum to physico-chemically precipitate metal ions into non-recyclable metal waste sludges. In addition chelating agents such as EDTA will not be required to bind and remove metal ions as is typically done in soil washing.

The main highlight of these experiments is, that they utilize inexpensive commercially available materials like HCl, and HNO_3.

6.4 RECOMMENDATIONS

We have already observed that cadmium can be extracted almost 100 percent by using very dilute concentrations of hydrochloric acid. But the lead needs the slightly higher concentration of acids for its maximum extraction.

A study can be done to selectively recover both lead and cadmium by slightly altering the above experimental procedures.

The cadmium can be extracted by running the column of contaminated beads using the above mentioned concentrations of 0.005 N HCl, 0.01 N HCl, 0.05 N HCl, 0.1 N HCl without any problem. But the lead can be extracted in higher magnitude by adding a more concentrated acid at the point where in no further lead can be extracted, say at about 350 ml where in the extraction of lead is almost zero. Add a higher concentration of acid at that point, and the remainder of lead will be extracted. This gives a separate peak to the lead thereby, extracting both the cadmium and lead will be observed on a graph by different peaks at different volumes. A point however to be noted that this would mean a large volume of the acid extraction and our basic idea is to concentrate the metal in the least volume so that it could be recycled economically.

A study could also be done regarding the contact time required between a particular strength of the acid to the maximum yield of extraction. This study may indicate that at a particular concentration of the acid taken knowing the approximate amount of yield extracted, the contact time will let us know the time required to get that particular yield.

Table 13

Mixture runs of cadmium and lead using 0.005 N HCl

Fraction #	Vol.eln.	% Lead	% Cadmium	Conc. of HCl in N
1.	25 ml	0.0	5.84	0.0
2.	25 ml	0.0	1.72	0.0
3.	25 ml	0.0	1.03	0.0
4.	25 ml	0.0	0.17	0.0
5.	25 ml	0.46	0.68	9.6×10^{-5}
6.	25 ml	1.86	5.16	2.4×10^{-4}
7.	25 ml	2.79	9.97	6.7×10^{-4}
8.	25 ml	2.98	11.00	1.2×10^{-3}
9.	25 ml	3.26	9.97	1.7×10^{-3}
10.	25 ml	3.58	8.94	2.3×10^{-3}
11.	25 ml	3.72	6.87	2.7×10^{-3}
12.	25 ml	4.00	4.81	3.0×10^{-3}
13.	25 ml	3.44	3.44	3.5×10^{-3}
14.	25 ml	2.79	2.75	4.0×10^{-3}
Total	350 ml	28.91	72.37	

Table 14

Mixture runs of Cadmium and Lead using 0.01 N HCl

Fraction #	Vol eln.	% lead	% Cadmium	Conc.of HCl in N
1	25 ml	0.46	7.22	0.0
2	25 ml	0.32	3.61	0.0
3	25 ml	0.23	1.72	0.0
4	25 ml	0.09	0.34	0.0
5	25 ml	0.84	1.37	3.0×10^{-4}
6	25 ml	2.84	8.25	7.0×10^{-4}
7	25 ml	3.02	10.66	2.0×10^{-3}
8	25 ml	3.07	12.72	3.0×10^{-3}
9	25 ml	3.68	11.69	3.8×10^{-3}
10	25 ml	3.86	9.63	4.9×10^{-3}
11	25 ml	4.05	8.39	5.6×10^{-3}
12	25 ml	4.28	6.19	6.8×10^{-3}
13	25 ml	3.26	5.50	8.0×10^{-3}
14	25 ml	2.37	3.44	8.8×10^{-3}
Total	350 ml	32.4	90.75	

Table 15

Mixture runs of cadmium and lead using 0.05 N HCl

Fraction #	Vol.eln.	% lead	% Cadmium	Conc.of HCl in N
1	25 ml	0.0	10.31	0.0
2	25 ml	0.0	3.44	0.0
3	25 ml	0.0	1.03	0.0
4	25 ml	0.0	0.17	0.0
5	25 ml	0.09	1.37	0.004
6	25 ml	1.44	10.66	0.011
7	25 ml	4.60	13.75	0.022
8	25 ml	6.05	15.81	0.034
9	25 ml	6.89	14.78	0.039
10	25 ml	7.22	8.94	0.044
11	25 ml	6.65	6.19	0.047
12	25 ml	6.49	0.17	0.050
13	25 ml	6.14	0.0	0.053
14	25 ml	0.04	0.0	0.055
Total	350 ml	45.66	86.64	

Table 16

Mixture runs of Cadmium and Lead using 0.1 N HCl

Fraction #	Vol.eln.	% lead	% Cadmium	Conc.of HCl in N
1	25 ml	0.0	5.5	0.0
2	25 ml	0.0	1.72	0.0
3	25 ml	0.0	1.03	0.0
4	25 ml	0.0	0.86	0.0
5	25 ml	0.0	3.09	0.002
6	25 ml	6.98	17.19	0.008
7	25 ml	7.26	19.25	0.017
8	25 ml	9.96	17.19	0.025
9	25 m	11.92	8.25	0.035
10	25 ml	9.31	6.87	0.045
11	25 ml	7.45	6.53	0.056
12	25 ml	6.15	4.47	0.064
13	25 ml	5.12	3.09	0.075
14	25 ml	4.65	1.37	0.082
Total	350 ml	68.83	96.44	

Table 17

Cumulative Percent metal recovered using diff.conc.of HCl

Fr.#	0.005 N			0.01 N		
	Pb	Cd	Pb/Cd	Pb	Cd	Pb/Cd
1	0.0	5.84	0.0	0.46	7.22	0.06
2	0.0	7.56	0.0	0.78	10.83	0.07
3	0.0	8.59	0.0	1.01	12.55	0.08
4	0.0	8.76	0.0	1.10	12.89	0.08
5	0.46	9.45	0.05	1.94	14.26	0.14
6	2.32	14.60	0.16	4.78	22.51	0.21
7.	5.12	24.57	0.20	7.80	33.17	0.23
8.	8.10	35.57	0.22	10.87	45.89	0.24
9.	11.36	45.55	0.25	14.55	57.58	0.25
10.	14.94	54.49	0.27	18.41	67.21	0.27
11.	18.67	61.36	0.30	22.46	75.60	0.29
12.	22.67	66.18	0.34	26.74	81.79	0.32
13.	26.11	69.62	0.37	30.00	87.29	0.34
14.	28.91	72.37	0.39	32.37	90.73	0.35

Table 18

Cumulative percent metal recovered using diff.conc.of HCl

Fr.#	0.05 N			0.10 N		
	Pb	Cd	Pb/Cd	Pb	Cd	Pb/Cd
1.	0.0	10.31	0.0	0.0	5.5	0.0
2.	0.0	13.75	0.0	0.0	7.22	0.0
3.	0.0	14.78	0.0	0.0	8.25	0.0
4.	0.0	14.95	0.0	0.0	9.11	0.0
5.	0.09	16.32	0.0	0.0	12.20	0.0
6.	1.53	26.98	0.05	6.98	29.39	0.24
7.	6.13	40.73	0.15	14.24	48.64	0.30
8.	12.18	56.54	0.21	24.20	65.83	0.36
9.	19.07	71.32	0.26	36.12	74.08	0.48
10.	26.29	80.26	0.32	45.43	80.95	0.56
11.	32.94	86.45	0.38	52.88	87.48	0.60
12.	39.43	86.62	0.45	59.03	91.95	0.64
13.	45.57	86.62	0.52	64.15	95.04	0.67
14.	45.61	86.62	0.53	68.80	96.41	0.71

BIBLIOGRAPHY.

1. Ioannis I. Valvis, "Biosynthesis of Amine Oxides using calcium-alginate immobilized hog liver microsomes: an evaluation of process and reactor design", Master thesis, NJIT, Newark, NJ.

2. Cornelius B. Murphy, Jr., Stuart J. Spiegel, " Biocaccumulation and toxicity of heavy metals and related trace elements ", Journal WPCF , Vol 54, # 6, pg 849, June 1982.

3. Y.P.Ting, F.Lawson, and I.G.Prince , " Uptake of cadmium and zinc by the alga chlorella vulgaris : part I. individual ion species ", Biotech. Bioeng., Vol 34, pg 990-999, 1989.

4. P.W.W. Kirk and J.N.Lester, " Significance and behaviour of heavymetals in wastewater treatment processes IV. Water quality criteria", The Science of Total Environment, Vol 40, pg 1-44, 1984.

5. Mohammad Ajmal and Ahsan Ullah Khan, " Effect of water hardness on the toxicity of cadmium to microorganisms ", Water Res. Vol 18, # 12, pg 1487-1491, 1984.

6. Anders B. Noreberg and Nils Molin, " Toxicity of cadmium, cobalt, uranium and zinc to zoologea ramigera ", Water Res. Vol 167, # 10, pg 1333-1336, 1983.

7. Remy Gourdon, Emilia Rus, Shubhangi Bhende, and S.S.Sofer,"Mechanism of cadmium uptake by activated sludge" Appl.Microbiol.Biotechnology, vol 34, pg 274-278, 1990.

8. R.M.Sterritt and J.N.Lester, "Aspects of the determination of complexation parameters for metal particulate complexes in activated sludge ", Water Res. Vol 19, # 3, pg 315-321, 1985.

9. Akira Nakajima and Takashi Sakaguchi, " Selective accumulation of heavy metals by microorganisms", Appl. Microbiol.Biotechnol, Vol 24 , pg 59-64, 1986.

10. R.M.Sterritt and J.N.Lester, "Interactions of heavy metals with bacteria", Science of Total Environment, Vol 14 pg 5-17, 1980.

11. W.P.Inverson and F.E.Brinckman, " Microbial metabolism of heavy metals", Water Pollution Microbiology, Vol 15, pg 201-232, 1978.

12. L.E.Macaskie, J.M.Wates, and A.C.R Dean, " Cadmium accumulation by a citrobacter sp. immobilized on gel and solid supports : applicability to the treatment of liquid wastes containing heavy metal cations ", Biotech. Bioeng., Vol 30, pg 66-73 , 1987.

13. Denise P. Higham, Peter J.Sadler and Michael D.Scawenk , " Cadmium resistance in pseudomanas putida ; growth and uptake of cadmium ", Journal of General Microbiology, Vol 131, pg 2539-2544, 1985.

14. C.f.Forster, " Factors involved in the settlement of activated sludge-II, the binding of polyvalent metals," Water Res. vol 19, # 10, pg 1265-1271, 1985.

15. L. Wong and J. G. Henry," Biological removal and chemical recovery of metals from sludges", Proceedings of 39th waste conference, Purdue Univ. May 1984.

16. J.A.Scott and S.J.Palmer," Sites of cadmium uptake in bacteria used for biosorption ", Appl. Microbiol.Biotechnol, Vol 33, pg 221-225, 1990.

17. Terry J.Logan, Robert E. Feltz," Effect of aeration, Cd concentration and solids content on acid extraction of Cd from a municipal wastewater sludge", Journal of WPCF vol.57 # 1,pg 406, May 1985.

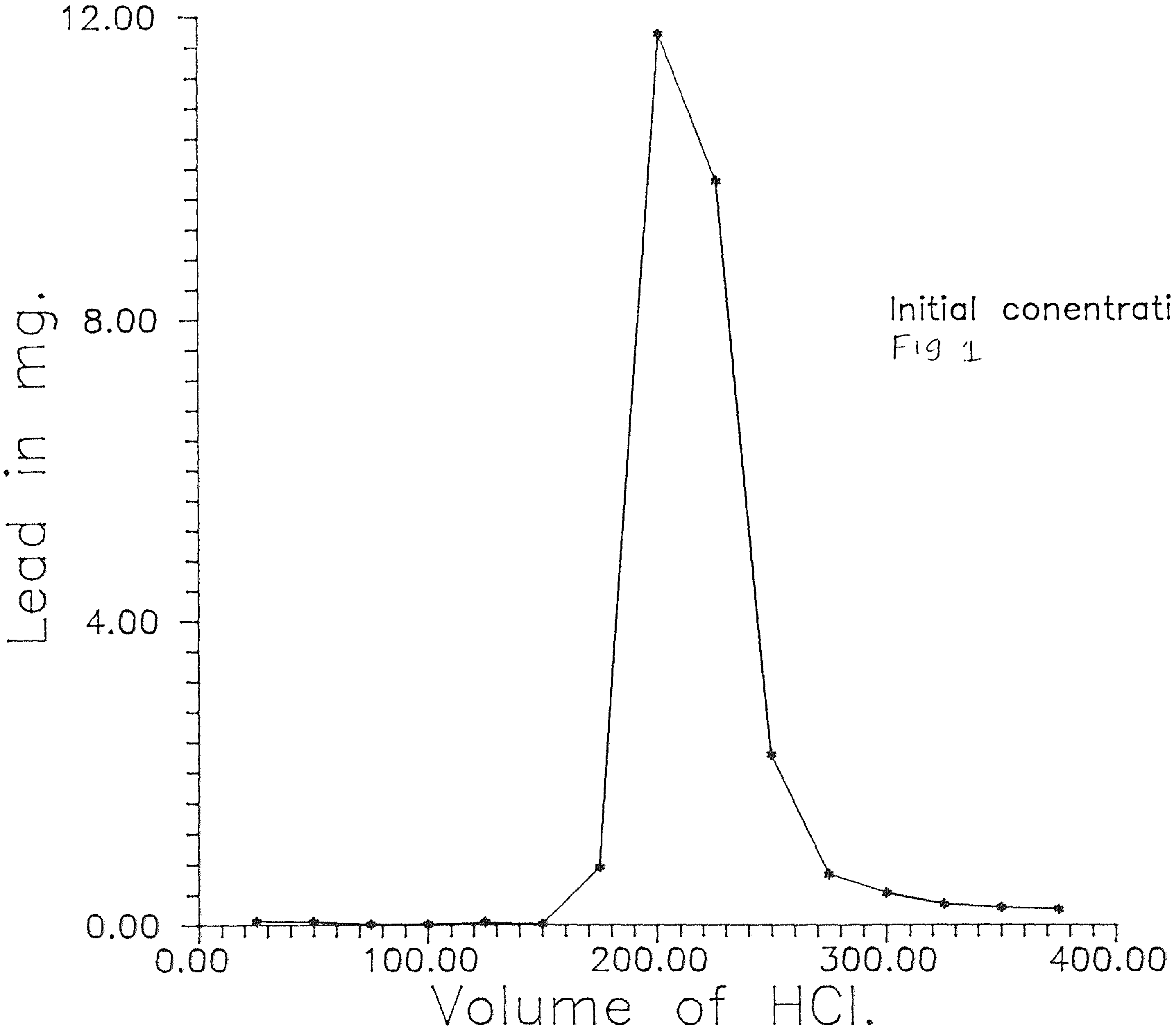
18. Melanie J.Brown and J.N.Lester, "Metal removal in activated sludge: The role of bacterial extra cellular polymers", Water Research Vol 13, pg 817-837, 1979.

19. John N.Lester, "Heavy metals in wastewater and sludge treatment processes", Treatment and Disposal, Vol 2, pg 21-30, 1979.

20 T.Rudd, R.M.Sterritt, and J.N.Lester,"Complexation of heavy metals by extra cellular polymers in the activated sludge process.", Journal WPCF, vol 56, # 12 , pg 1260, Dec. 1984.

21. Rainer C. Kistier and Fritz Widmer, " Behaviour of chromium, nickel, copper, zinc, cadmium, mercury, and lead during the pyrolysis of sewage sludge.", Environ.Sci. Technol., vol 21, # 7, pg 704, 1987.

22. Shoou-yuh Chang, Ju-Chang Huang and Yow-Chyun Liu,
" Effects of Cd (III) and Cu (II) on a biofilm system ",
Journal of Environmental Engineering, Vol 112, # 1, pg 94,
Feb. 1986



Initial conentration is 0.1 HCl.
Fig 1

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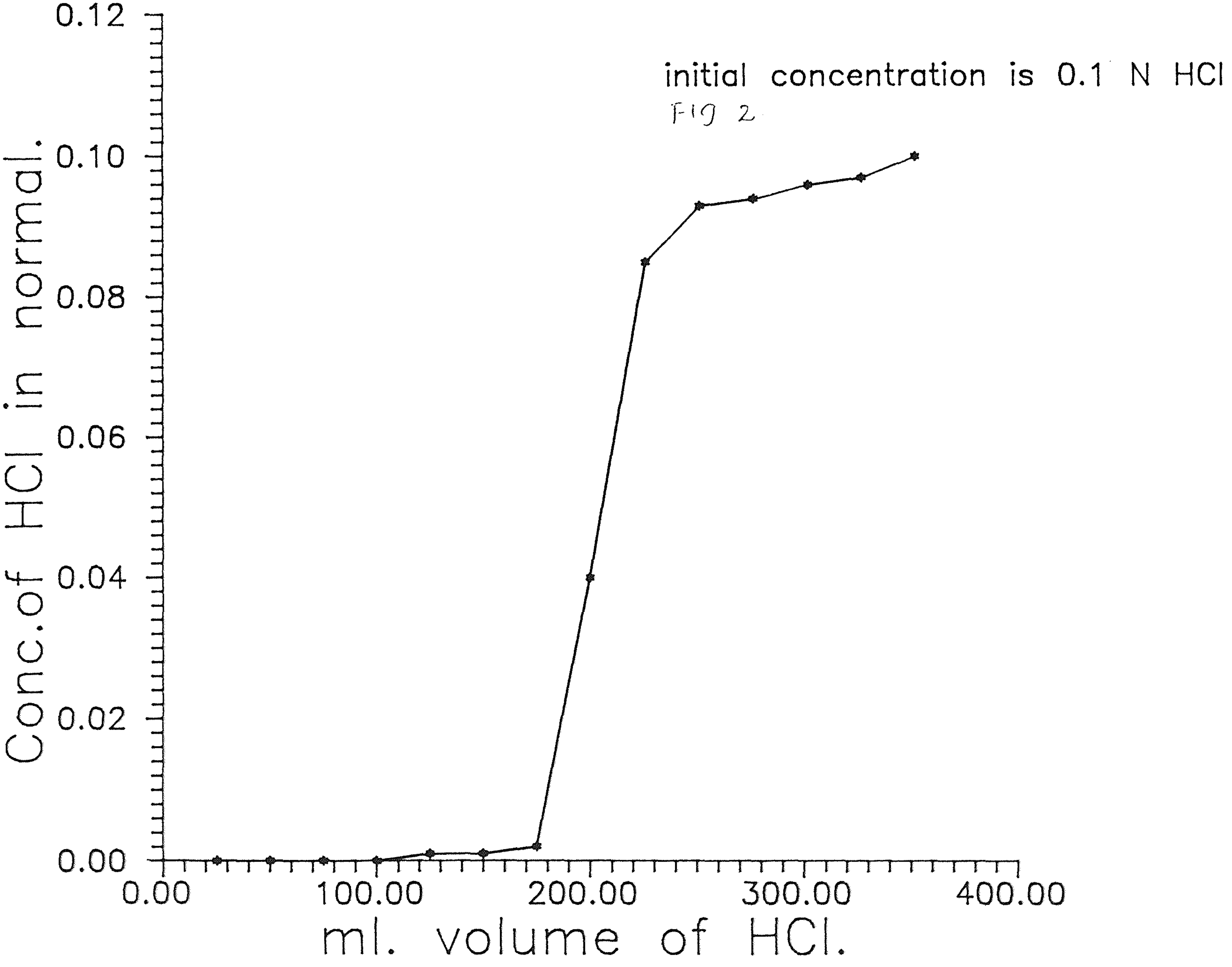
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Conc.of HCl in normal.
0.12
0.10
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100.00
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400.00
ml. volume of HCl.

FIG 2

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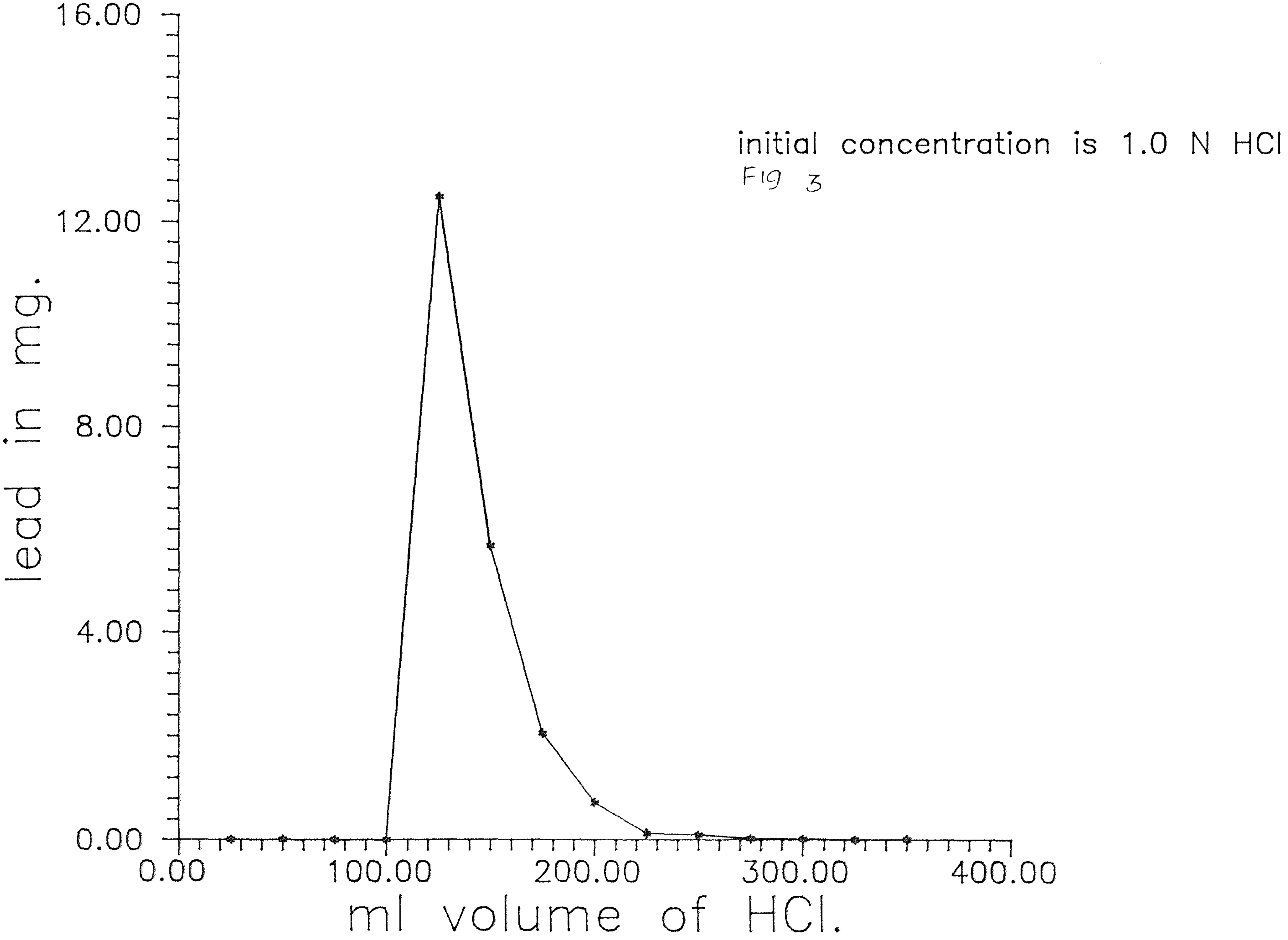

initial concentration is 1.0 N HCl

Fig 3

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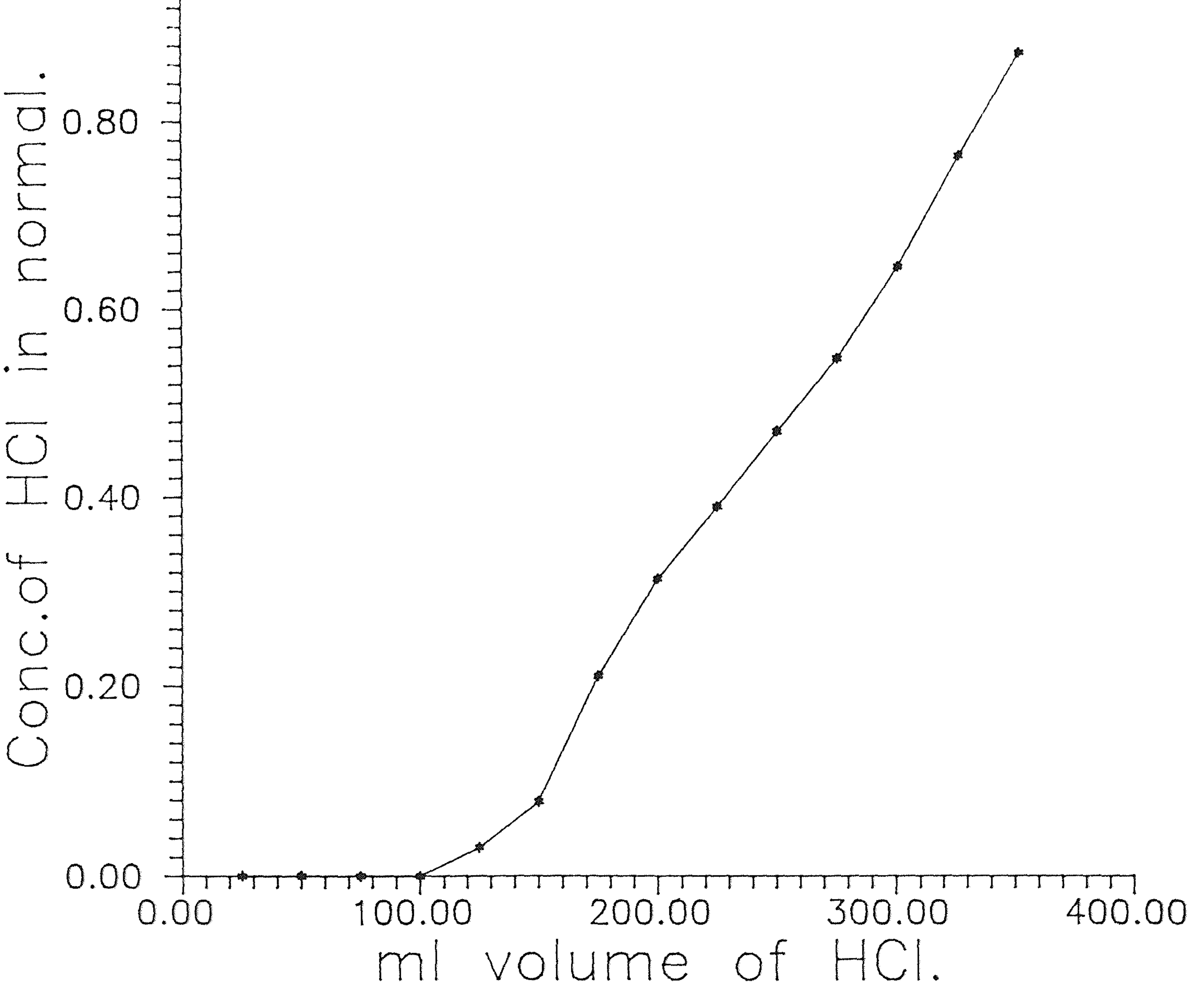
initial concn.is 1.0 N HCl

Fig 4



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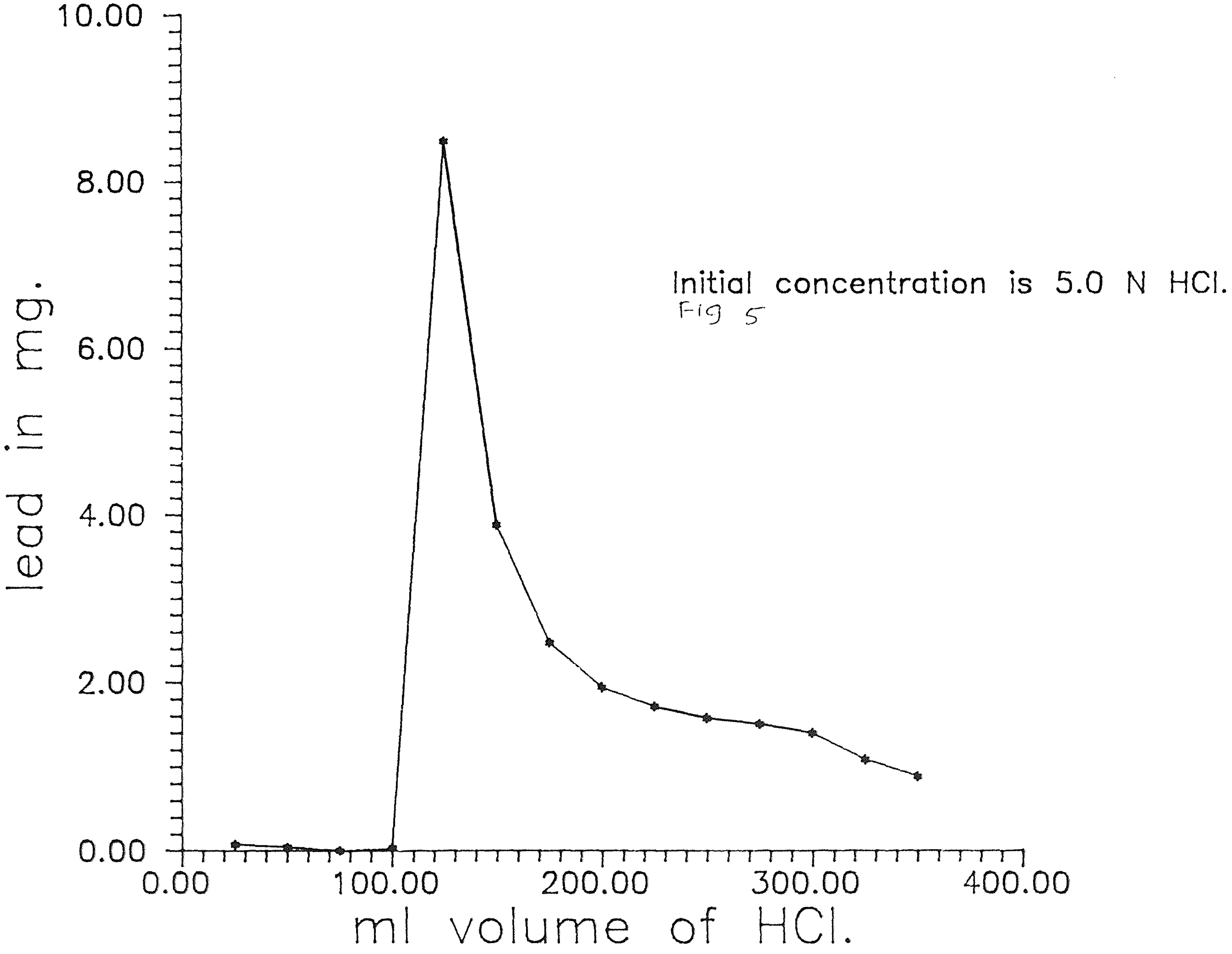
Initial concentration is 5.0 N HCl.
lead in mg.
10.00
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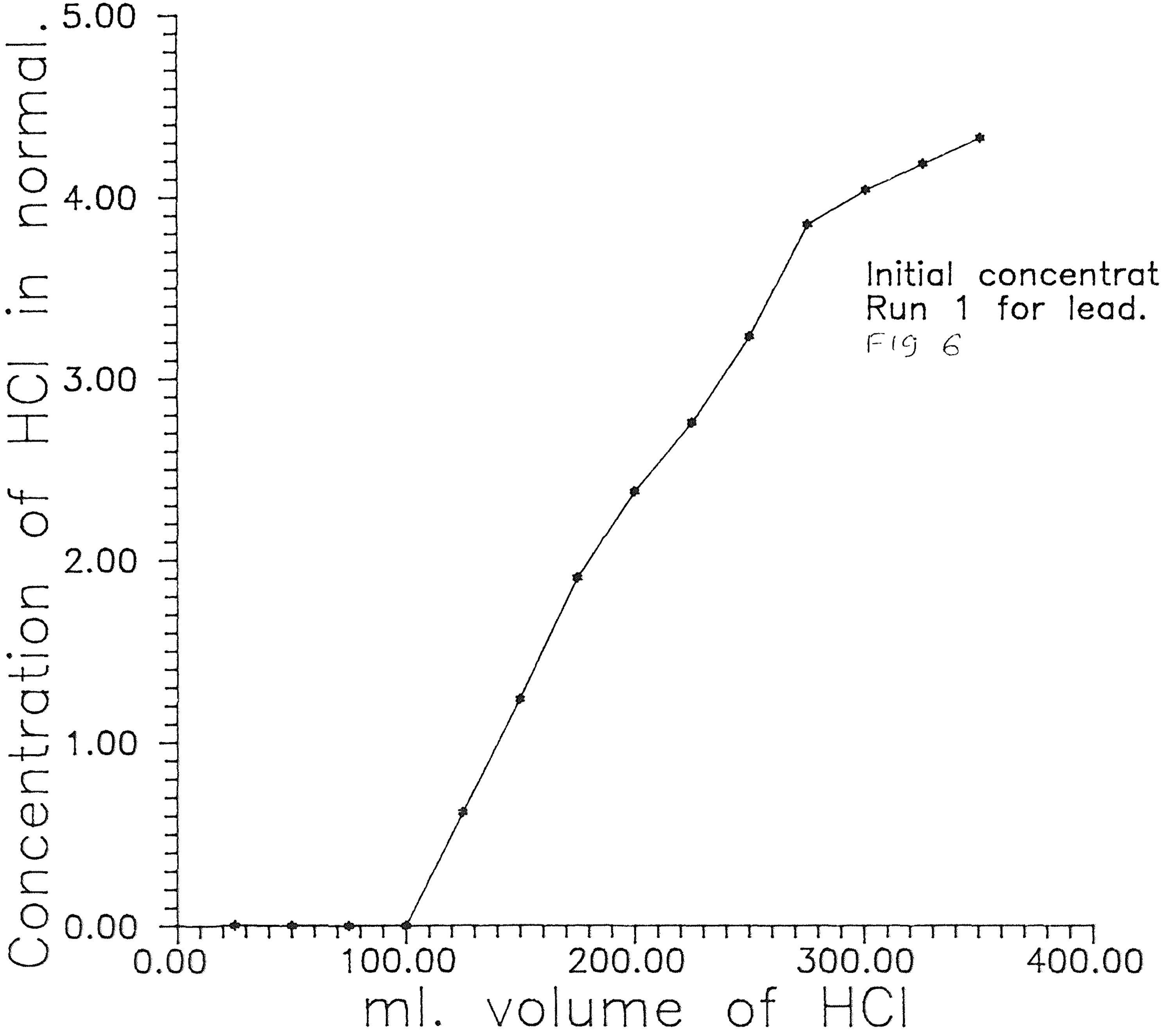
0.00
0.00
100.00
200.00
300.00
400.00
ml volume of HCl.

Fig 5



Initial concentration is 5.0 N HCl
Run 1 for lead.
FIG 6

~~[illegible]~~ 6A
(01

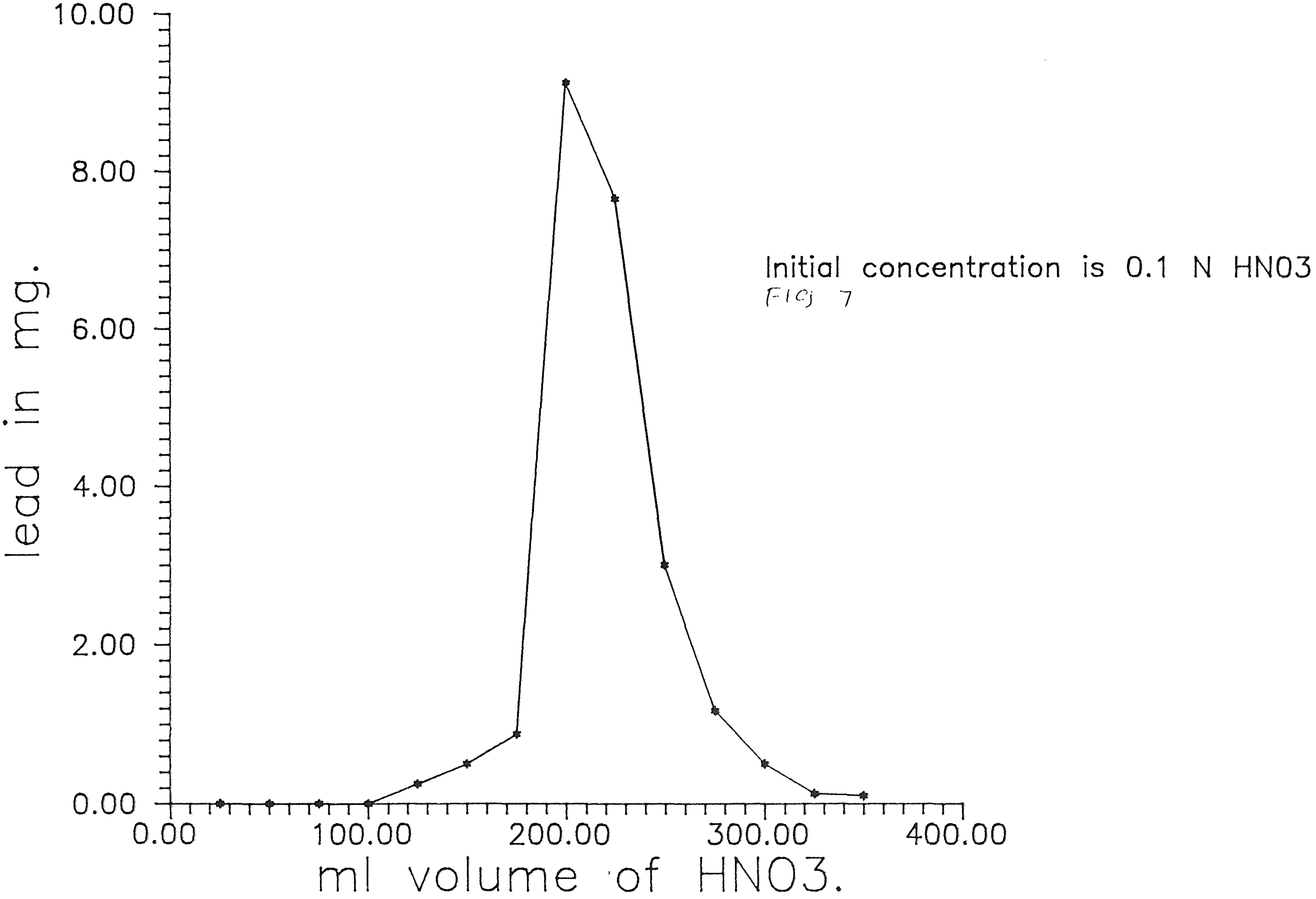

10.00
8.00
6.00
4.00
2.00
0.00
lead in mg.
0.00
100.00
200.00
300.00
400.00
ml volume of HNO3.
Initial concentration is 0.1 N HN03

FIG 7

Titan

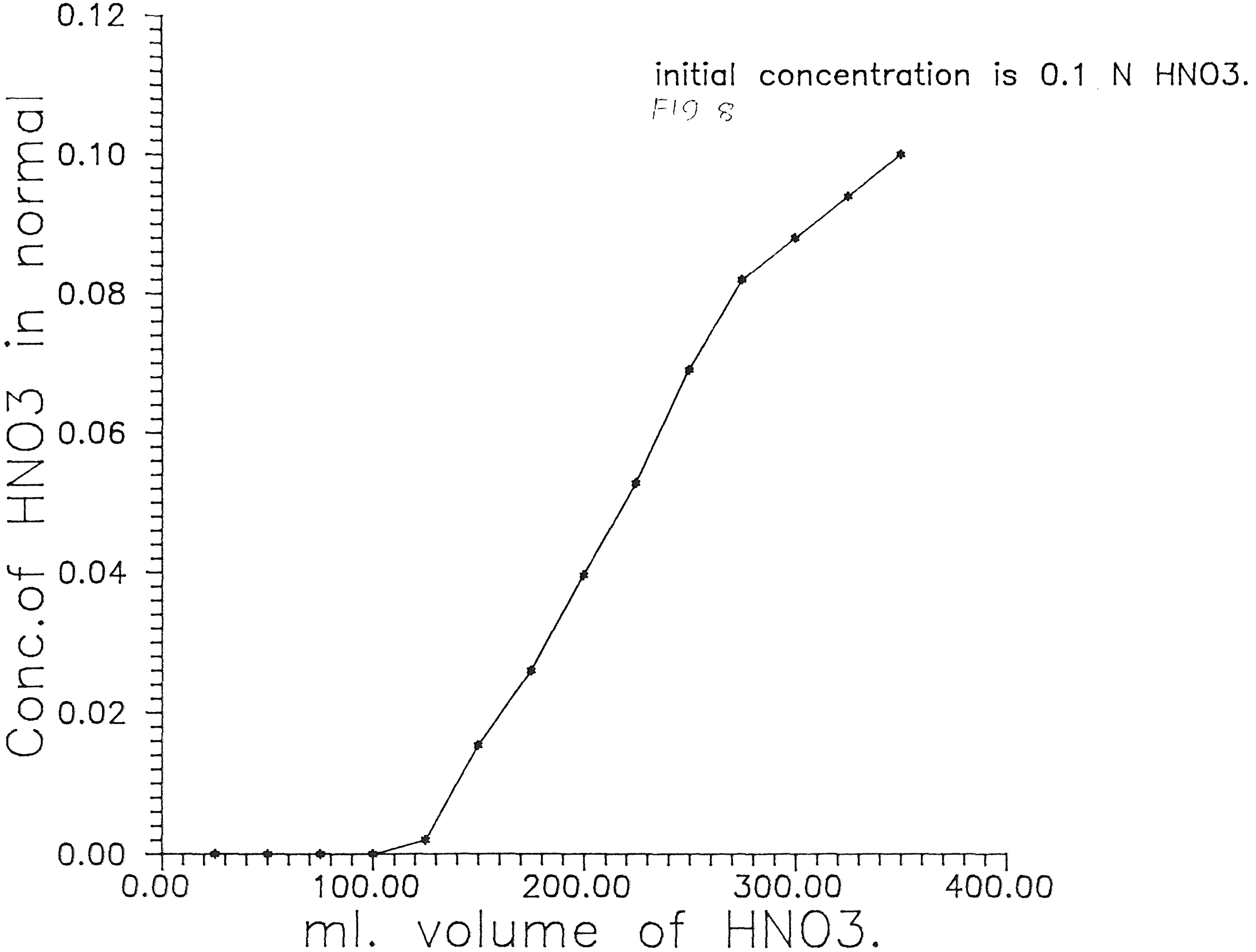

initial concentration is 0.1 N HNO3.
Conc.of HNO3 in normal
0.12
0.10
0.08
0.06
0.04
0.02
0.00
0.00
100.00
200.00
300.00
400.00
ml. volume of HNO3.

FIG 8

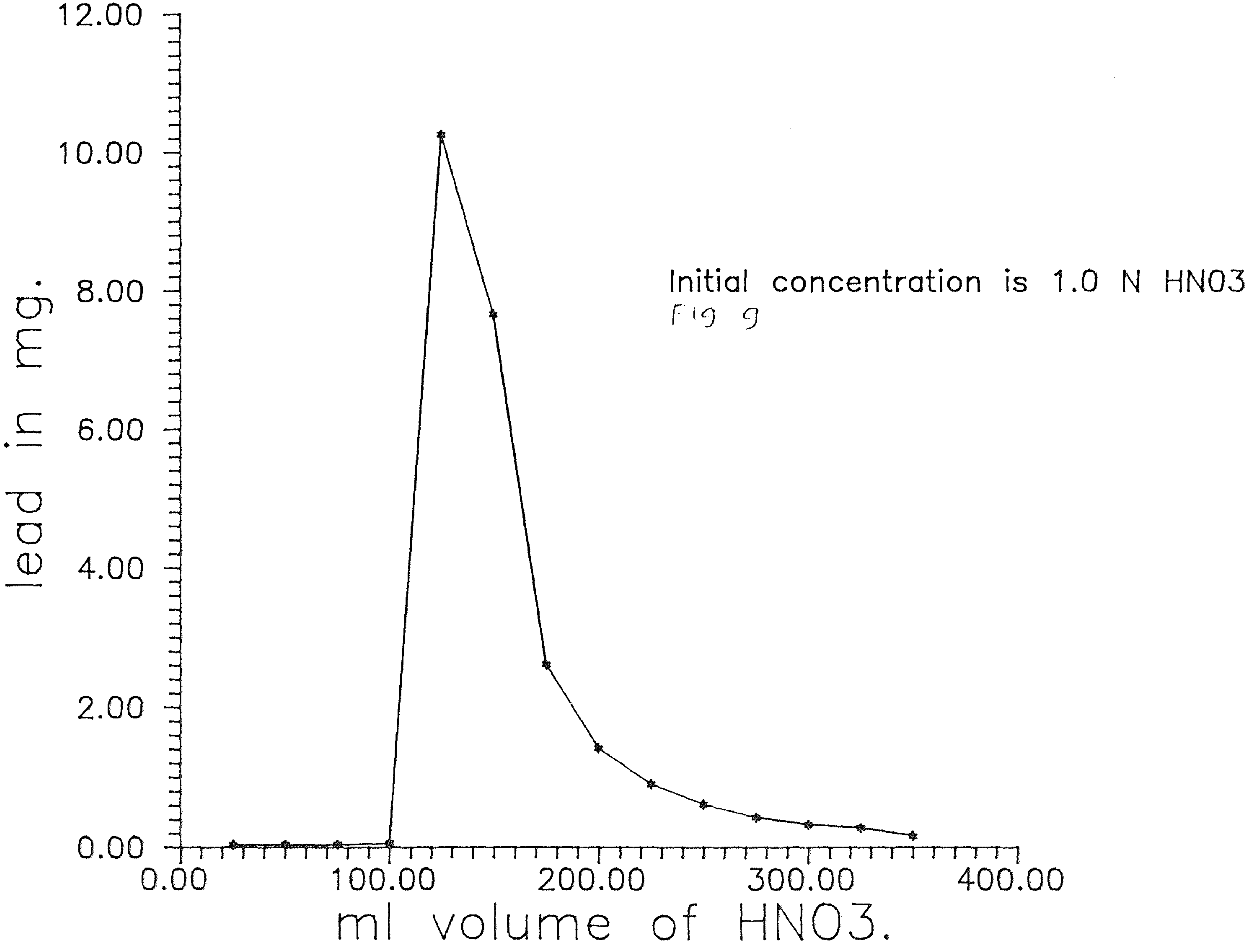

Initial concentration is 1.0 N HN03
lead in mg.
12.00
10.00
8.00
6.00
4.00
2.00
0.00
0.00
100.00
200.00
300.00
400.00
ml volume of HNO3.

Fig 9

Ti:F2N

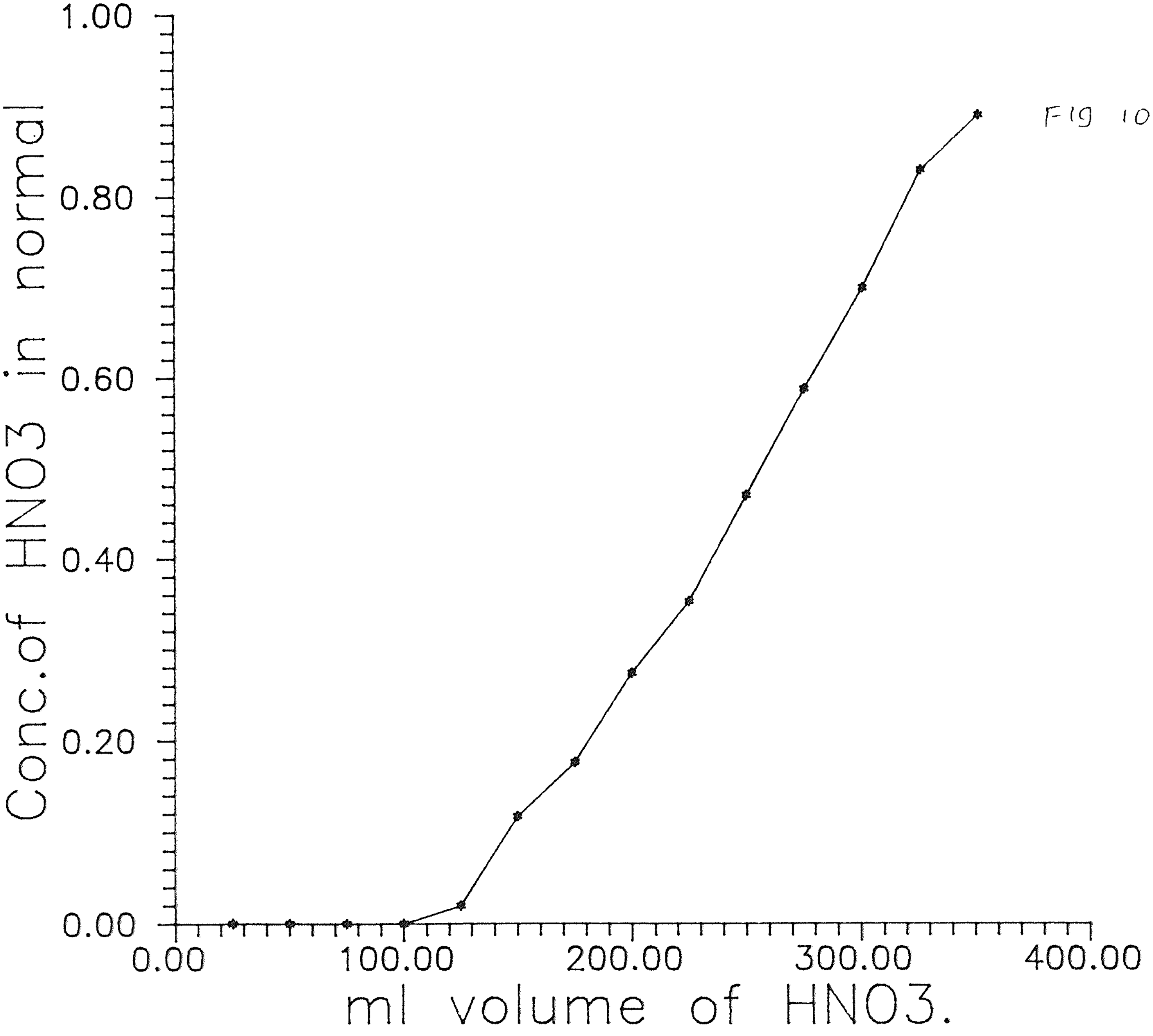

1.00
0.80
0.60
0.40
0.20
0.00
Conc.of HNO3 in normal
0.00
100.00
200.00
300.00
400.00
ml volume of HNO3.

Fig 10

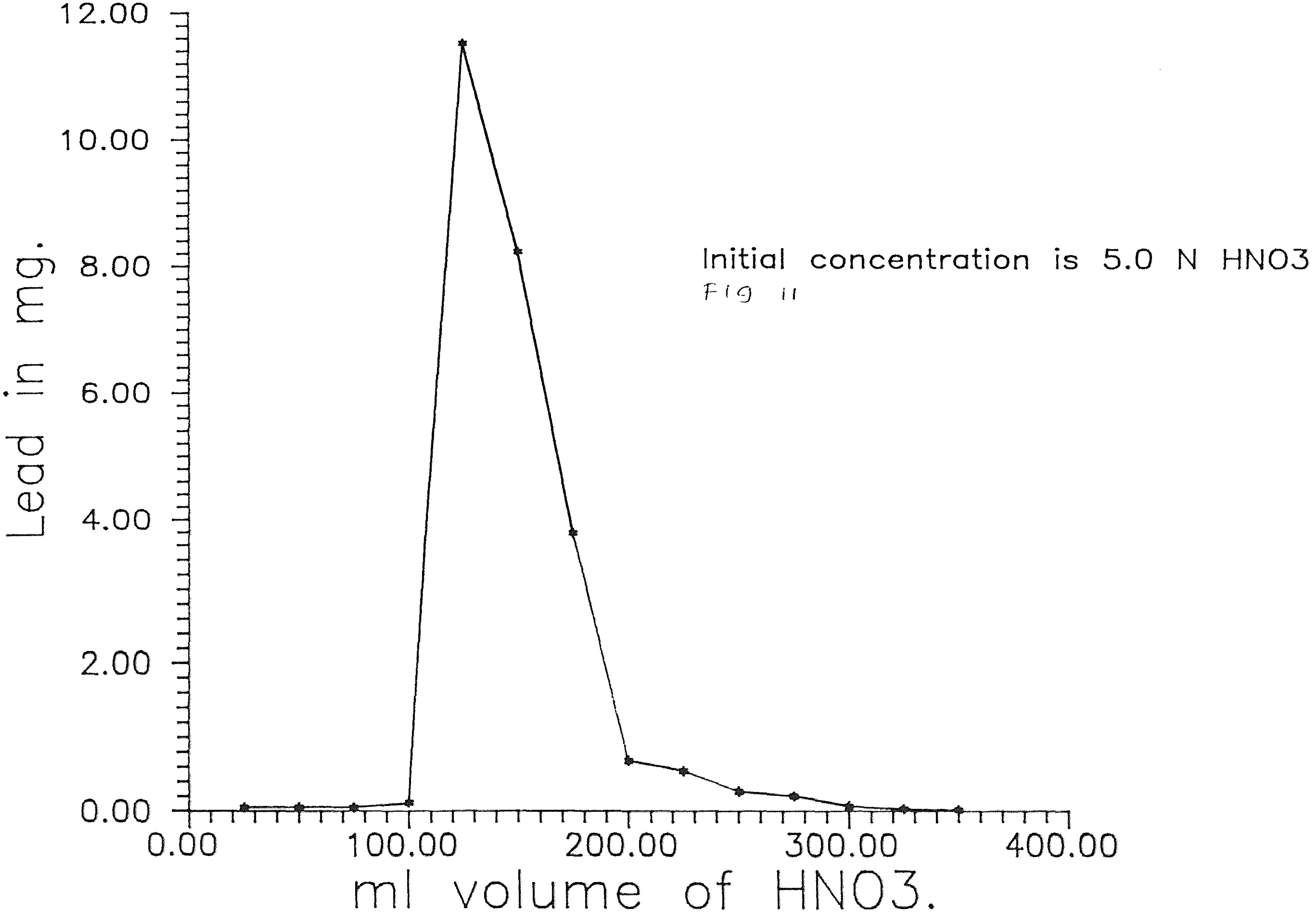

Initial concentration is 5.0 N HNO3
Lead in mg.
12.00
10.00
8.00
6.00
4.00
2.00
0.00
0.00
100.00
200.00
300.00
400.00
ml volume of HNO3.

Fig 11

Tit3N

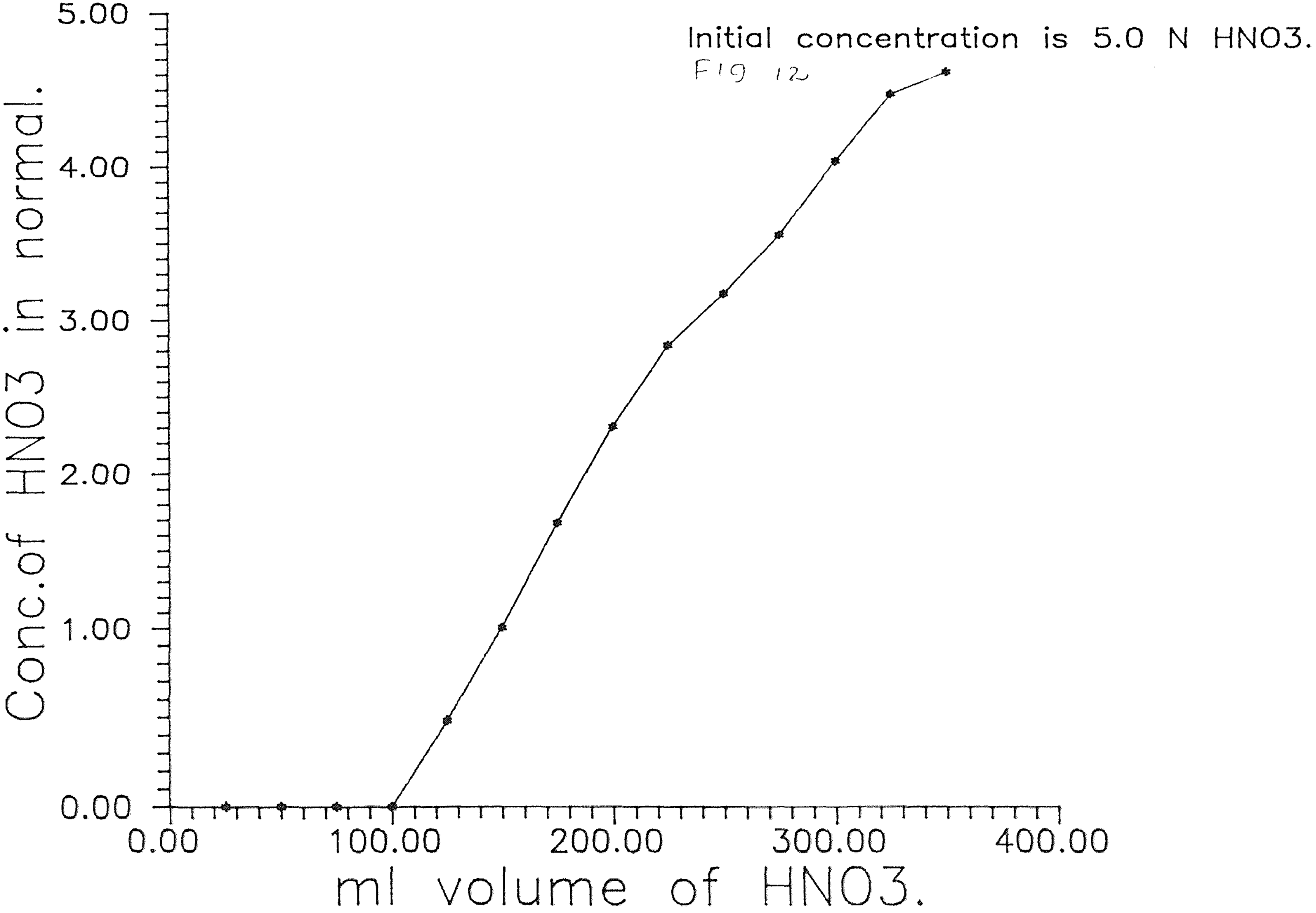

Initial concentration is 5.0 N HNO3.
5.00
4.00
3.00
2.00
1.00
0.00
Conc.of HNO3 in normal.
0.00
100.00
200.00
300.00
400.00
ml volume of HNO3.

Fig 12

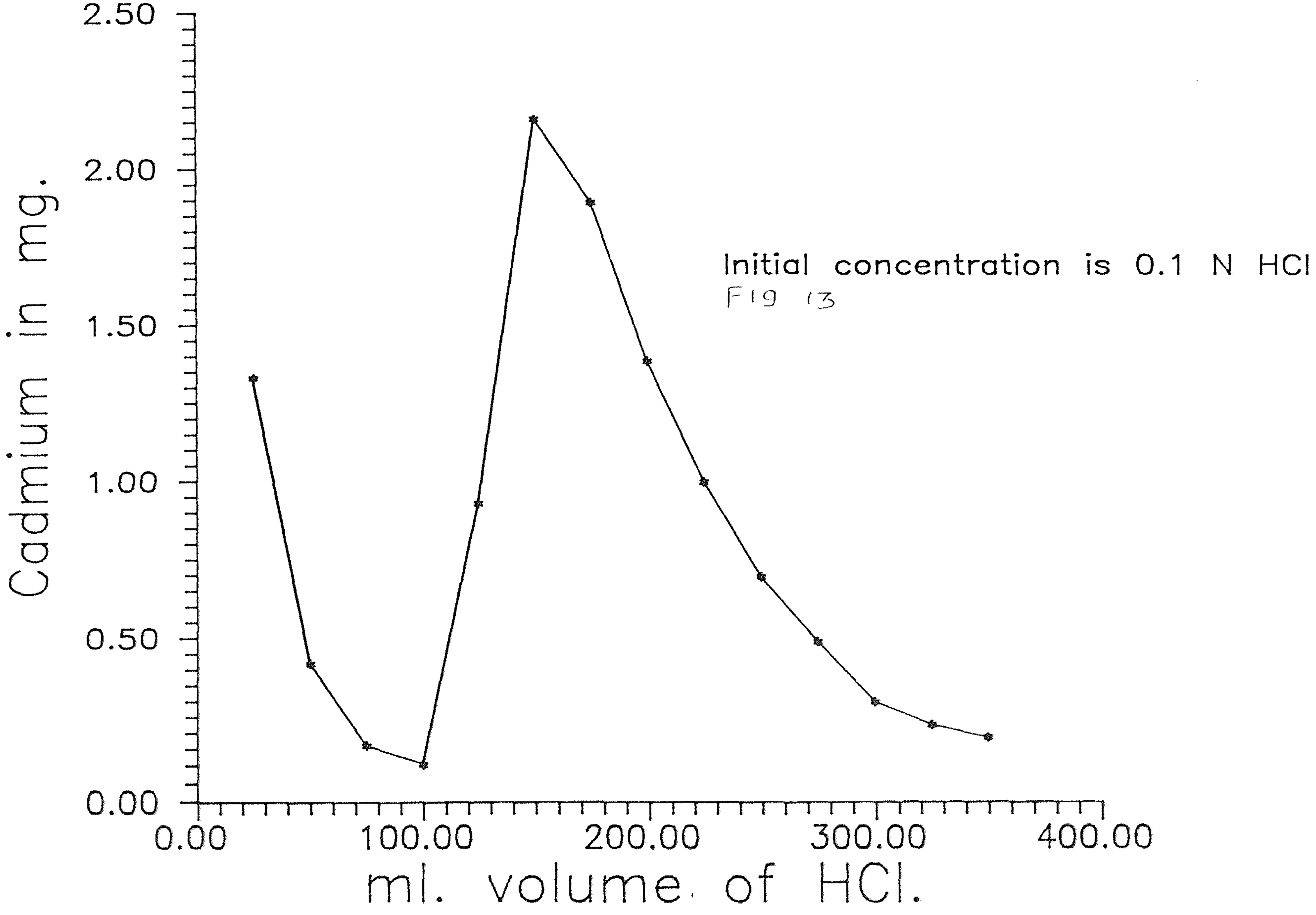
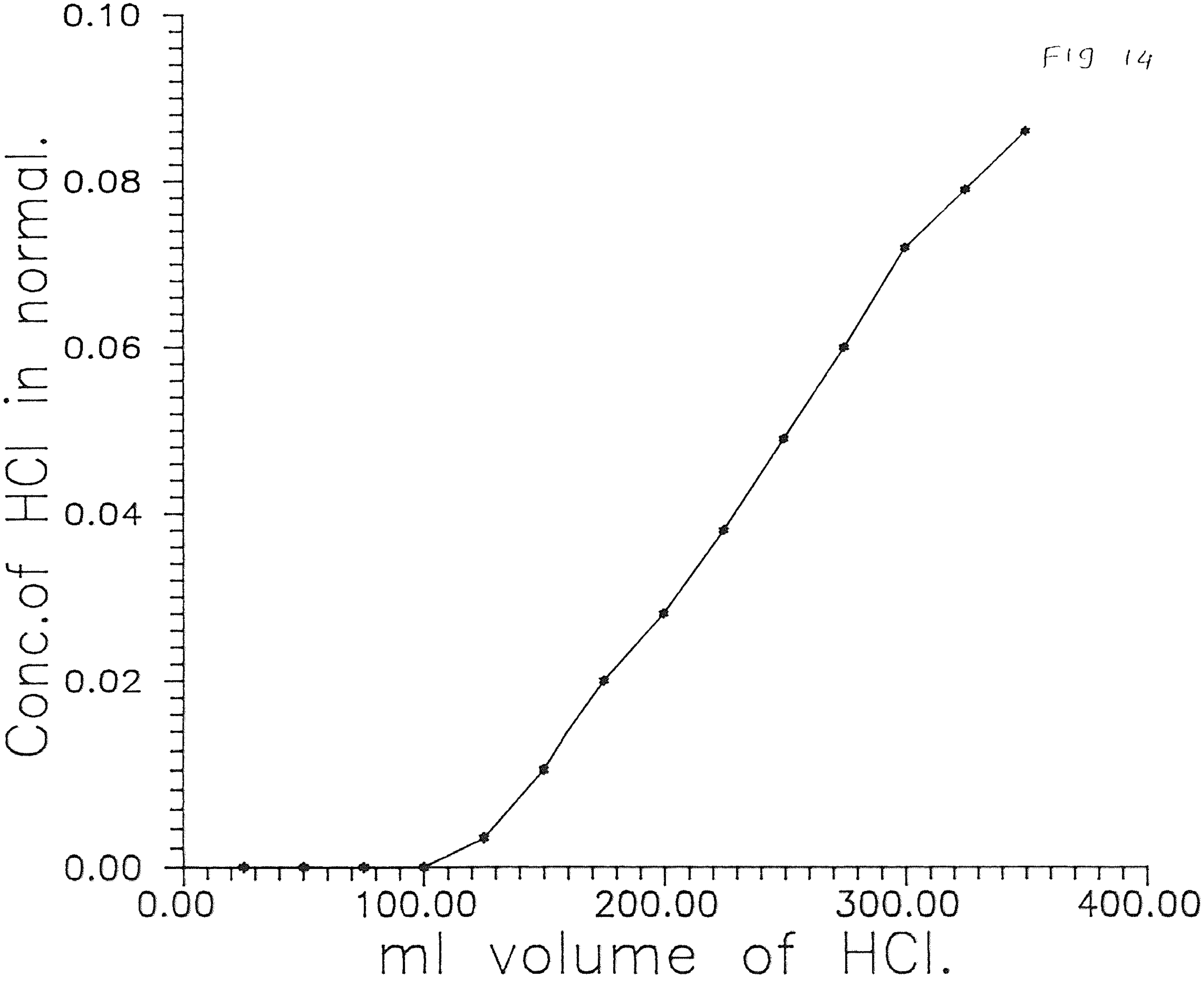

Initial concentration is 0.1 N HCl
Cadmium in mg.
2.50
2.00
1.50
1.00
0.50
0.00
0.00
100.00
200.00
300.00
400.00
ml. volume of HCl.

Fig 13

TITC2

Fig 14


Conc.of HCl in normal.
0.10
0.08
0.06
0.04
0.02
0.00
0.00
100.00
200.00
300.00
400.00
ml volume of HCl.

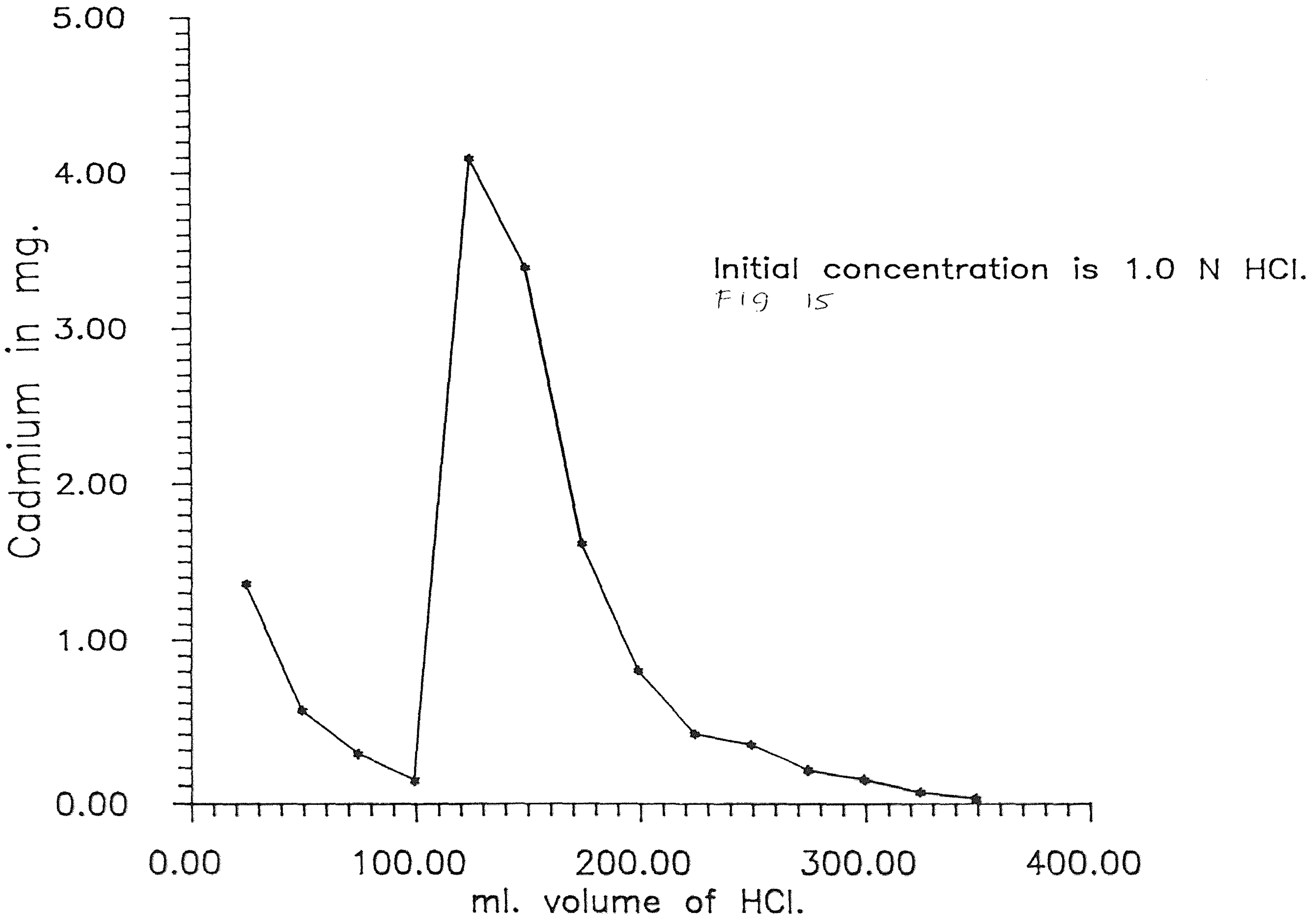

Initial concentration is 1.0 N HCl.
Cadmium in mg.
5.00
4.00
3.00
2.00
1.00
0.00
0.00
100.00
200.00
300.00
400.00
ml. volume of HCl.

FIG 15

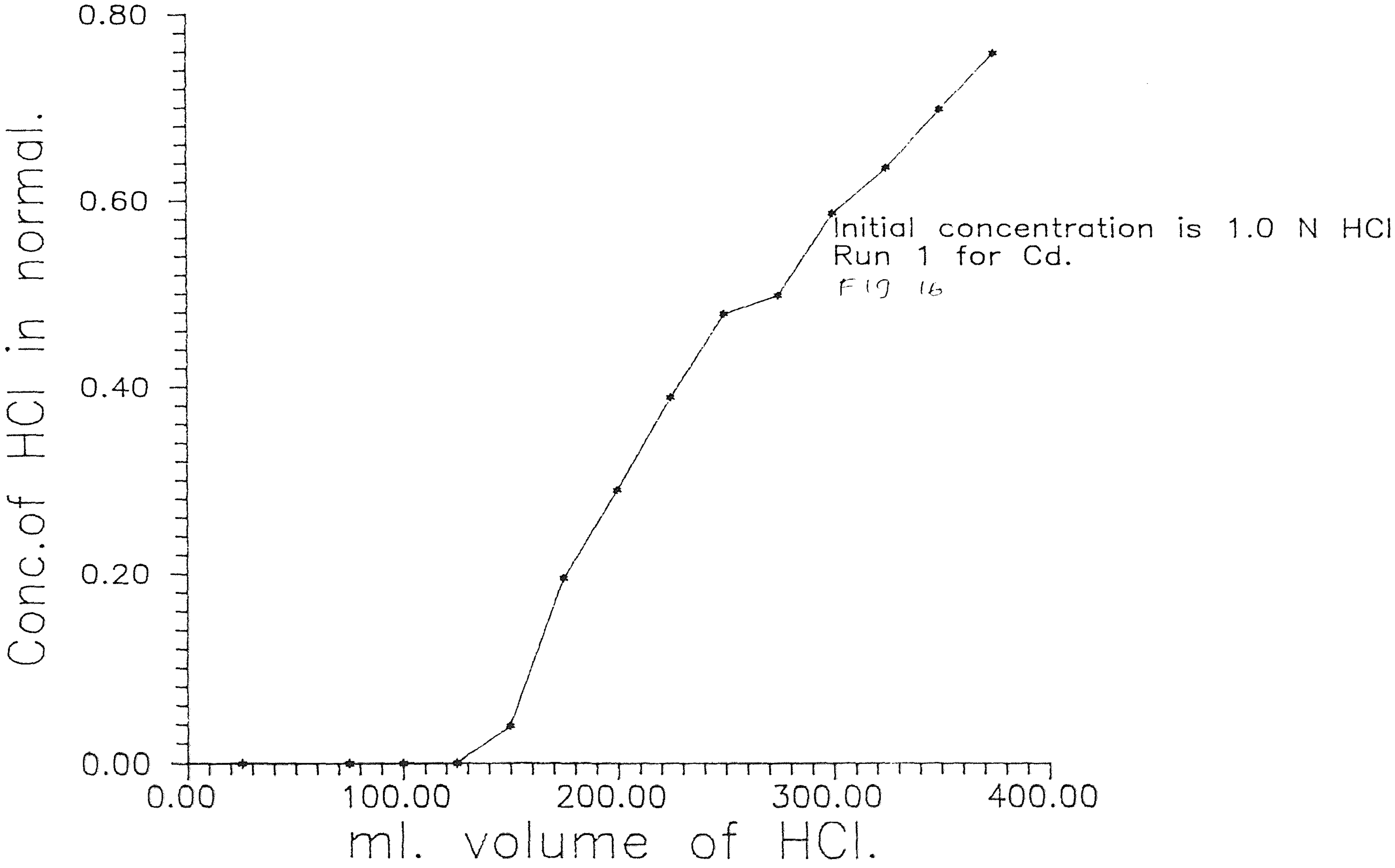

Initial concentration is 1.0 N HCl
Run 1 for Cd.
0.80
0.60
0.40
0.20
0.00
0.00
100.00
200.00
300.00
400.00
Conc.of HCl in normal.
ml. volume of HCl.

FIG 16

File name cd39.

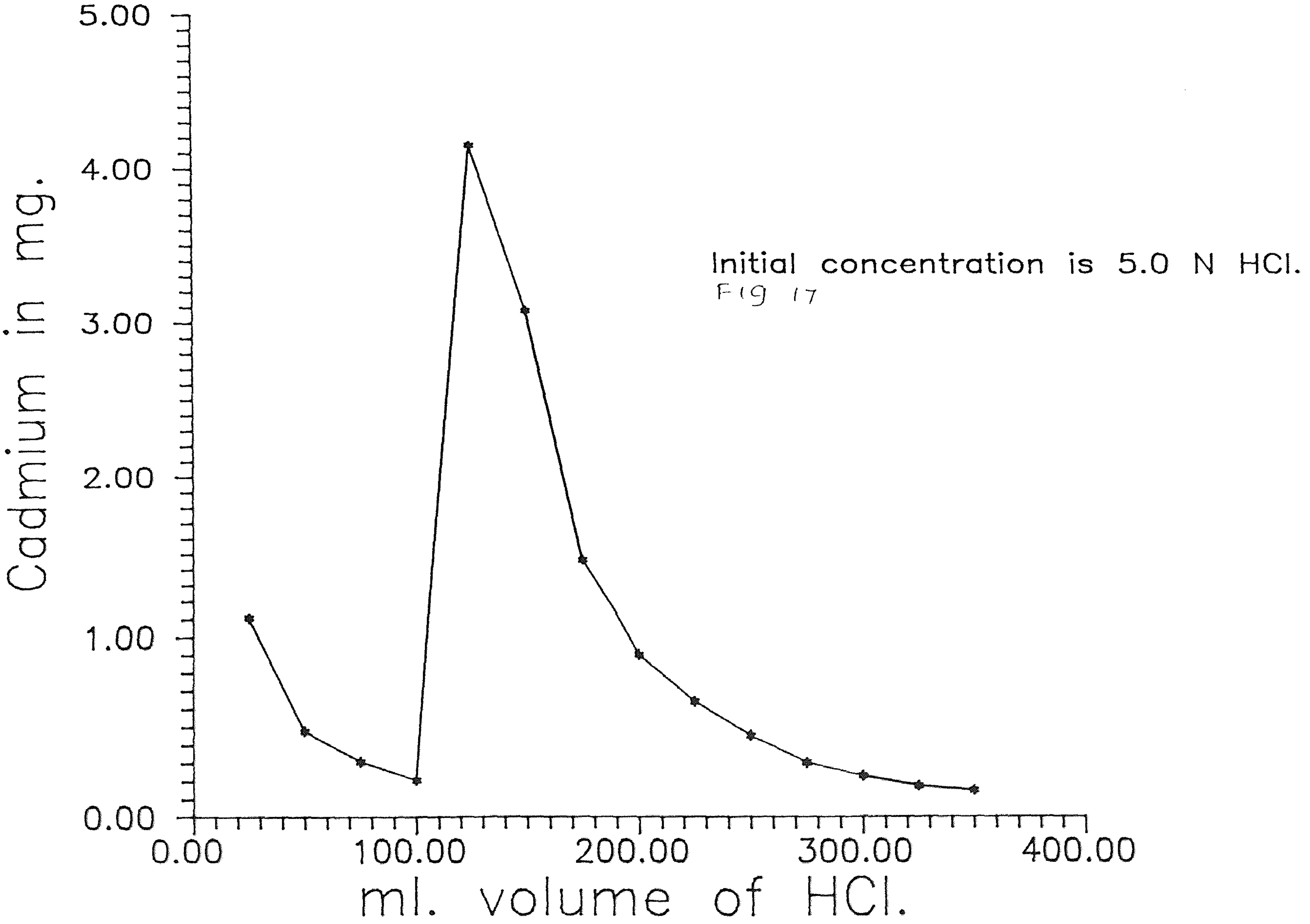


FIG 17

Tit 5a.
5N HCl

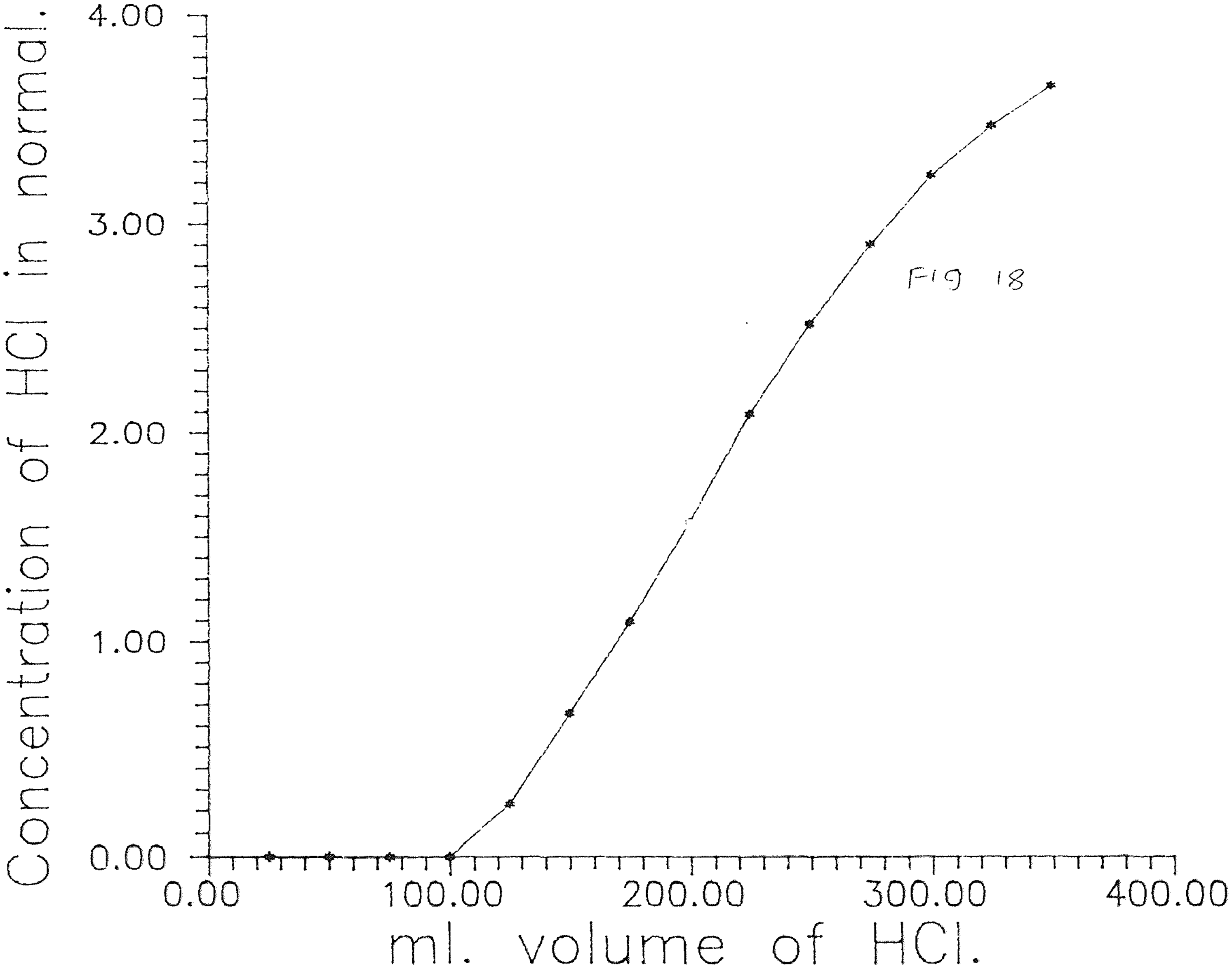


Fig 18

casa

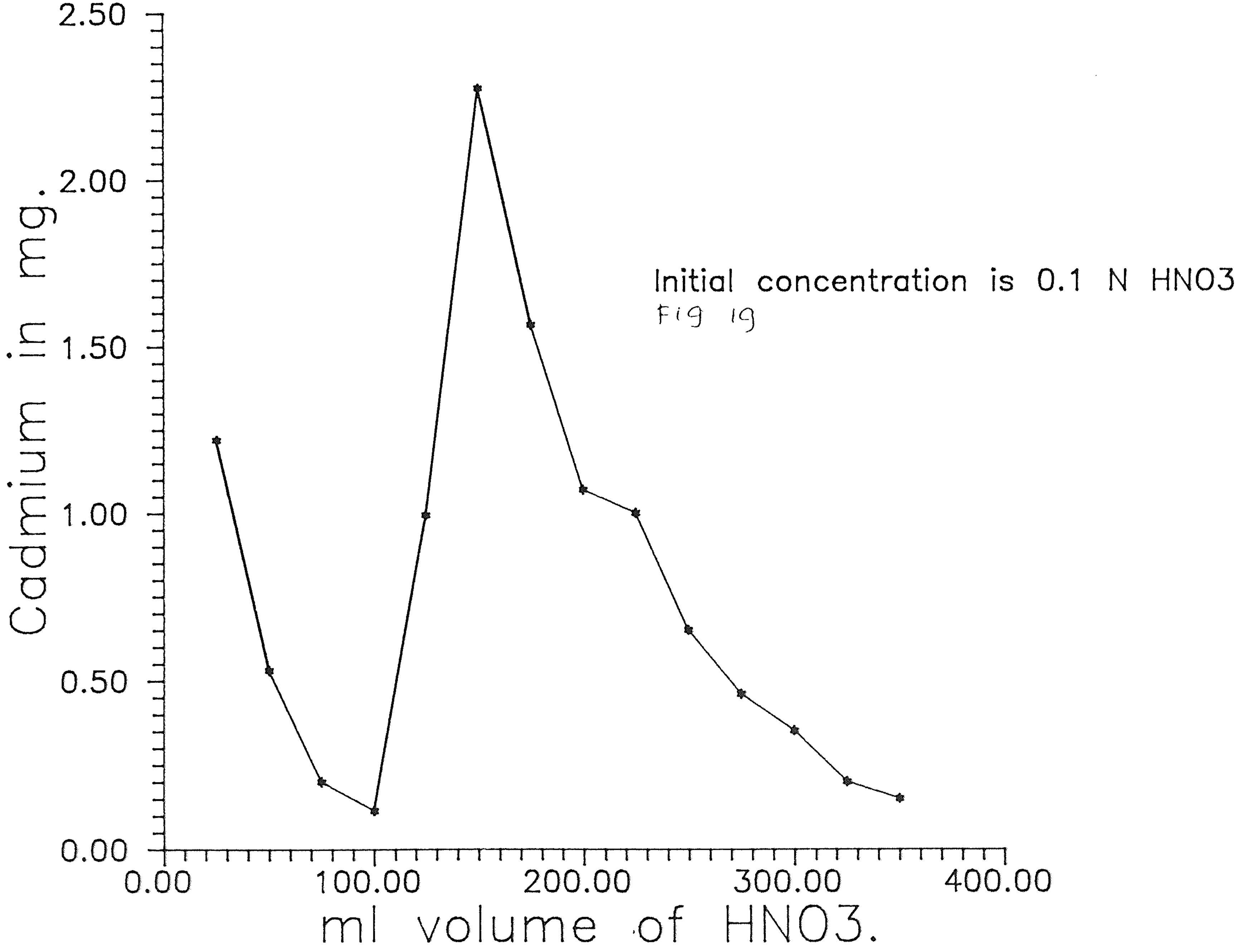

Initial concentration is 0.1 N HNO3
Cadmium in mg.
2.50
2.00
1.50
1.00
0.50
0.00
0.00
100.00
200.00
300.00
400.00
ml volume of HNO3.

Fig 19

Tit 6N

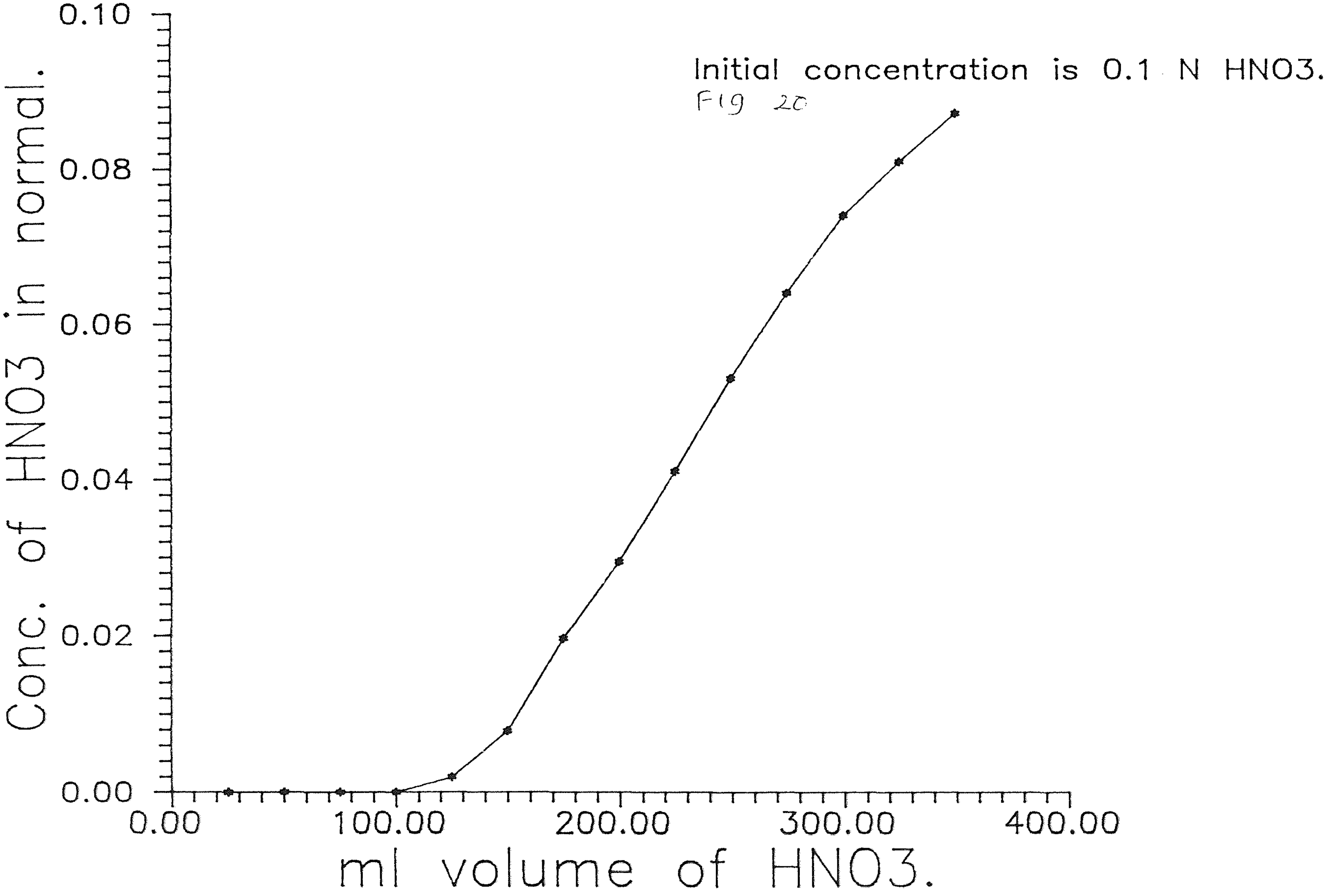
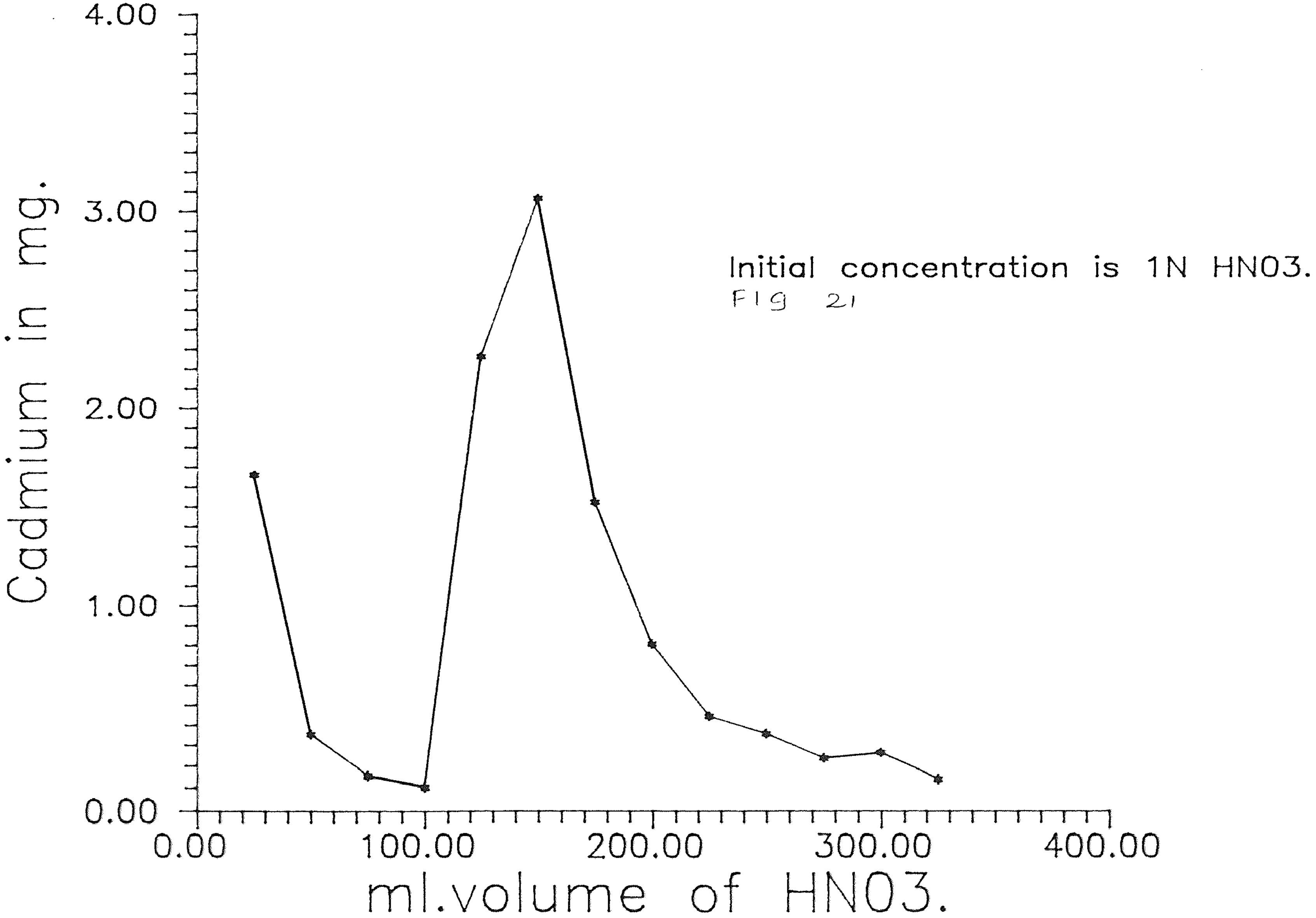

Initial concentration is 0.1 N HNO3.
Conc. of HNO3 in normal.
0.10
0.08
0.06
0.04
0.02
0.00
0.00
100.00
200.00
300.00
400.00
ml volume of HNO3.

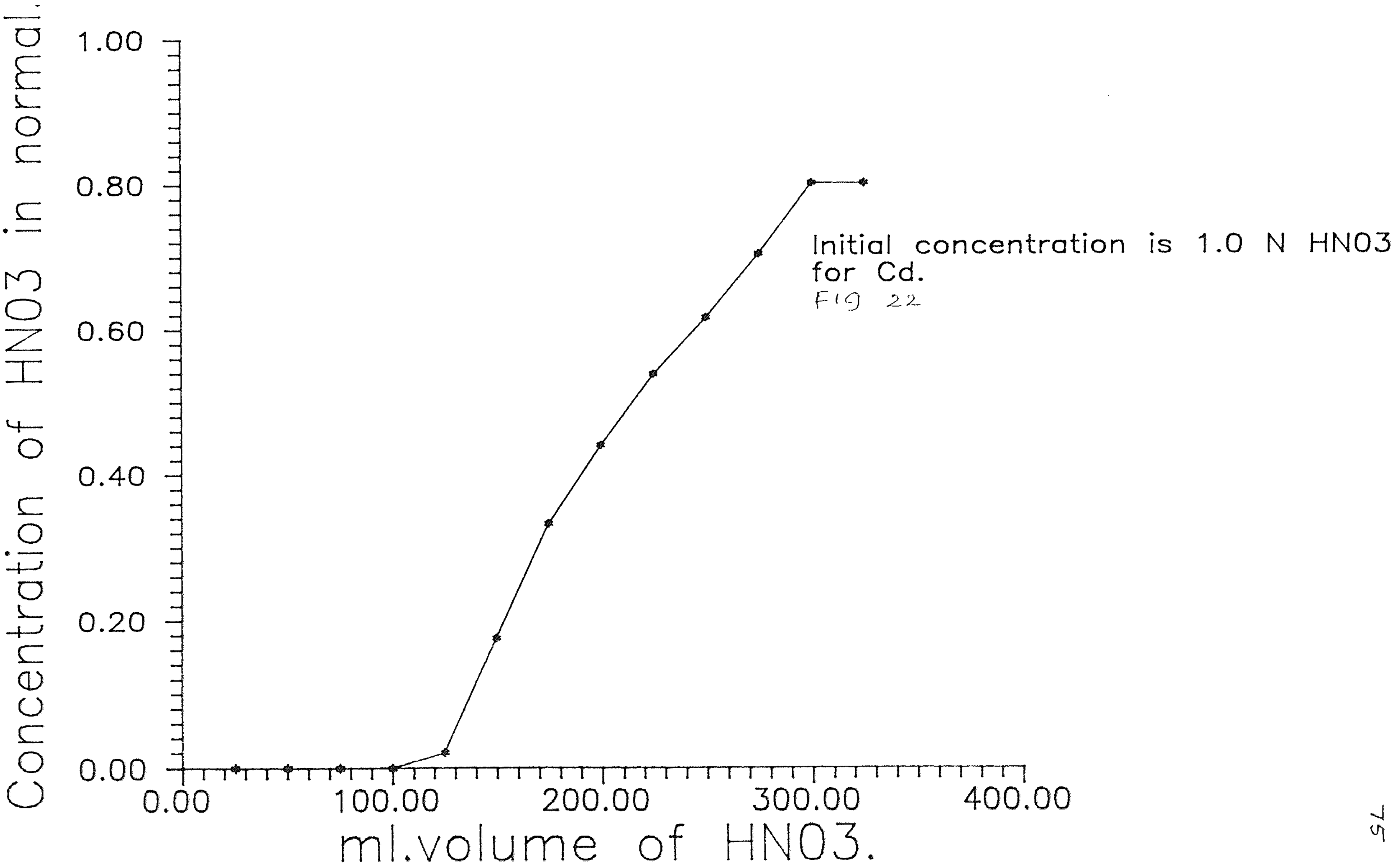
Fig 20

Cd4a



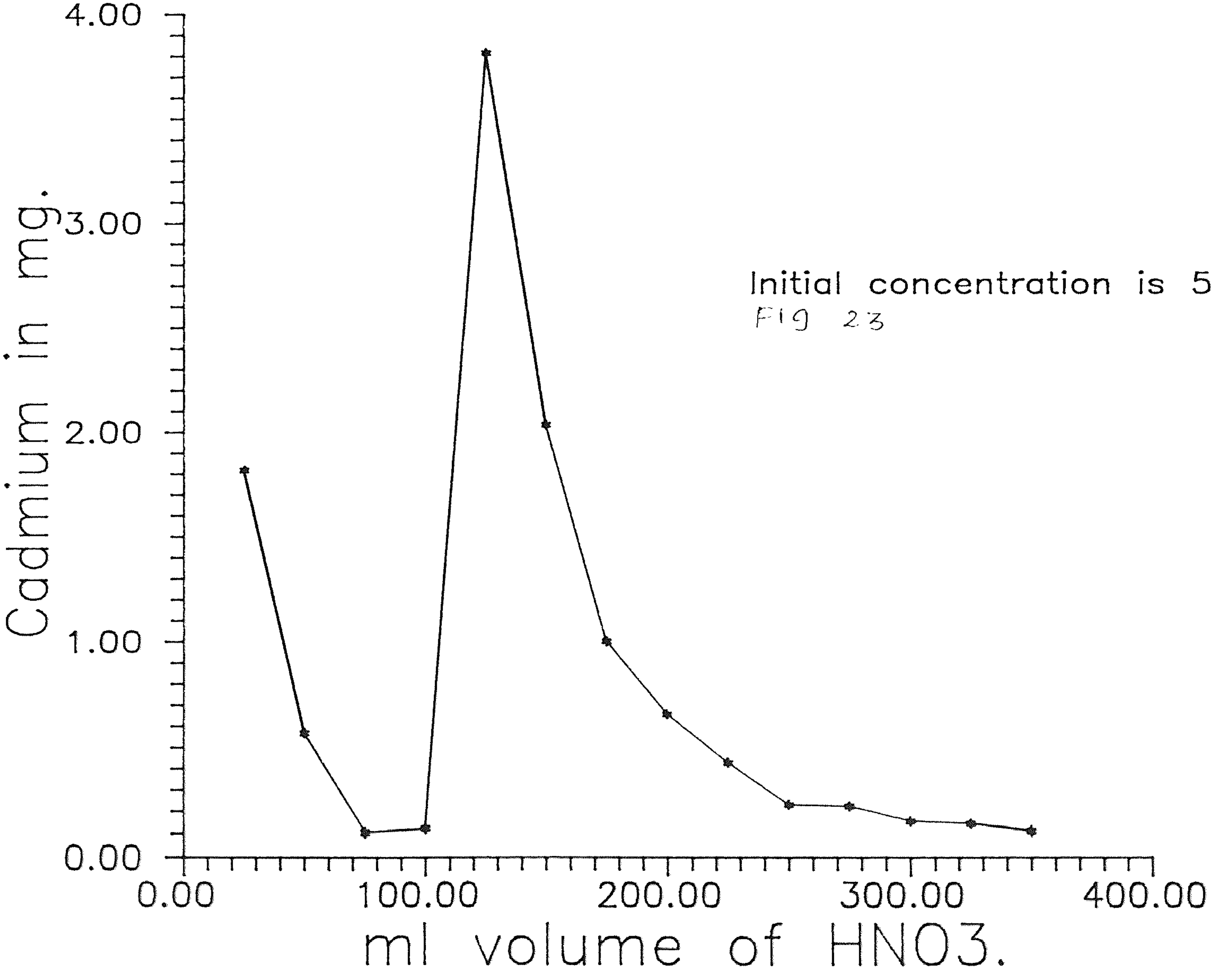
Initial concentration is 1N HN03.

FIG 21



Initial concentration is 1.0 N HN03
for Cd.

FIG 22



Initial concentration is 5.0 N HNO3.

FIG 23

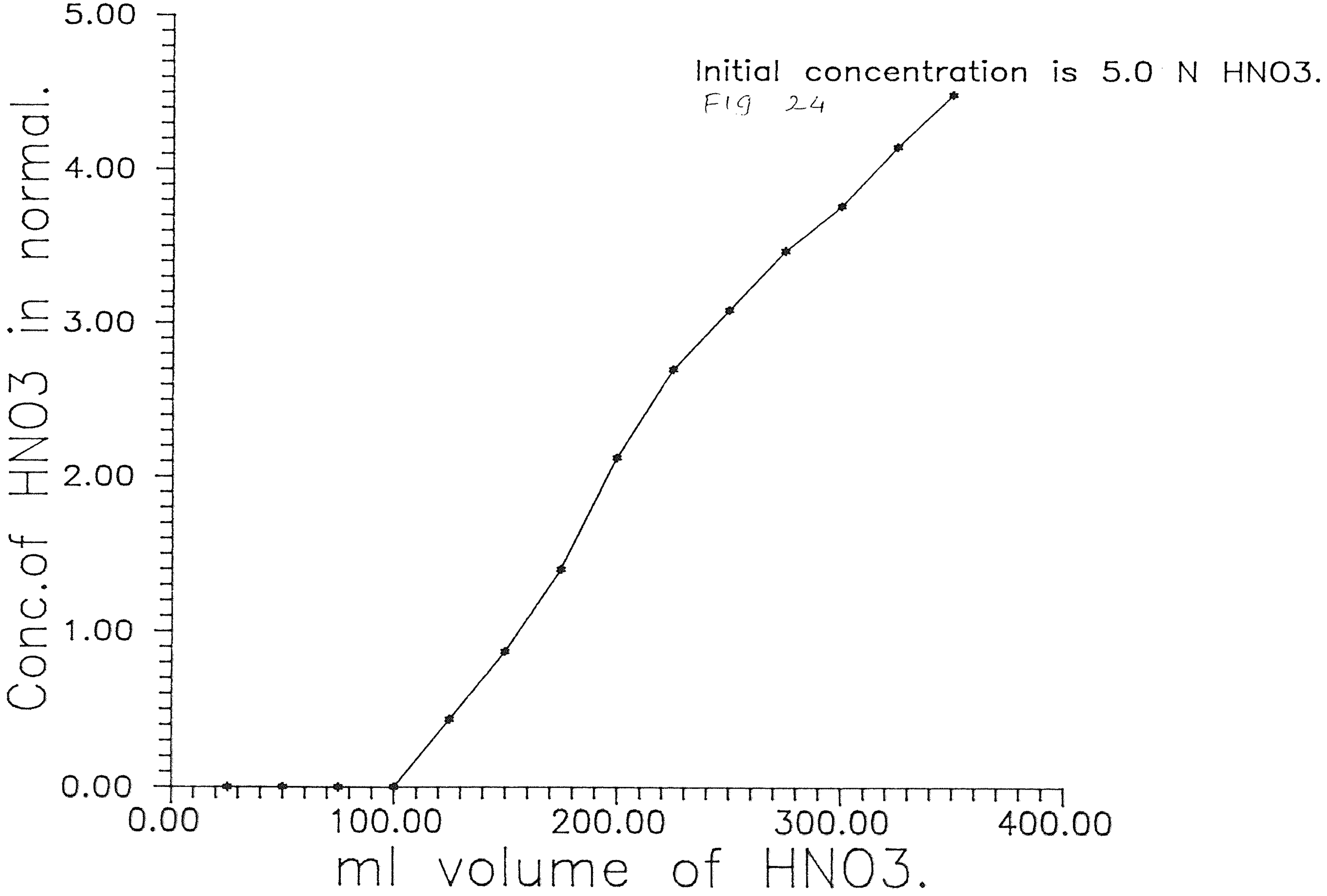

Initial concentration is 5.0 N HNO3.
5.00
4.00
3.00
2.00
1.00
0.00
Conc.of HNO3 in normal.
0.00
100.00
200.00
300.00
400.00
ml volume of HNO3.

FIG 24

A1

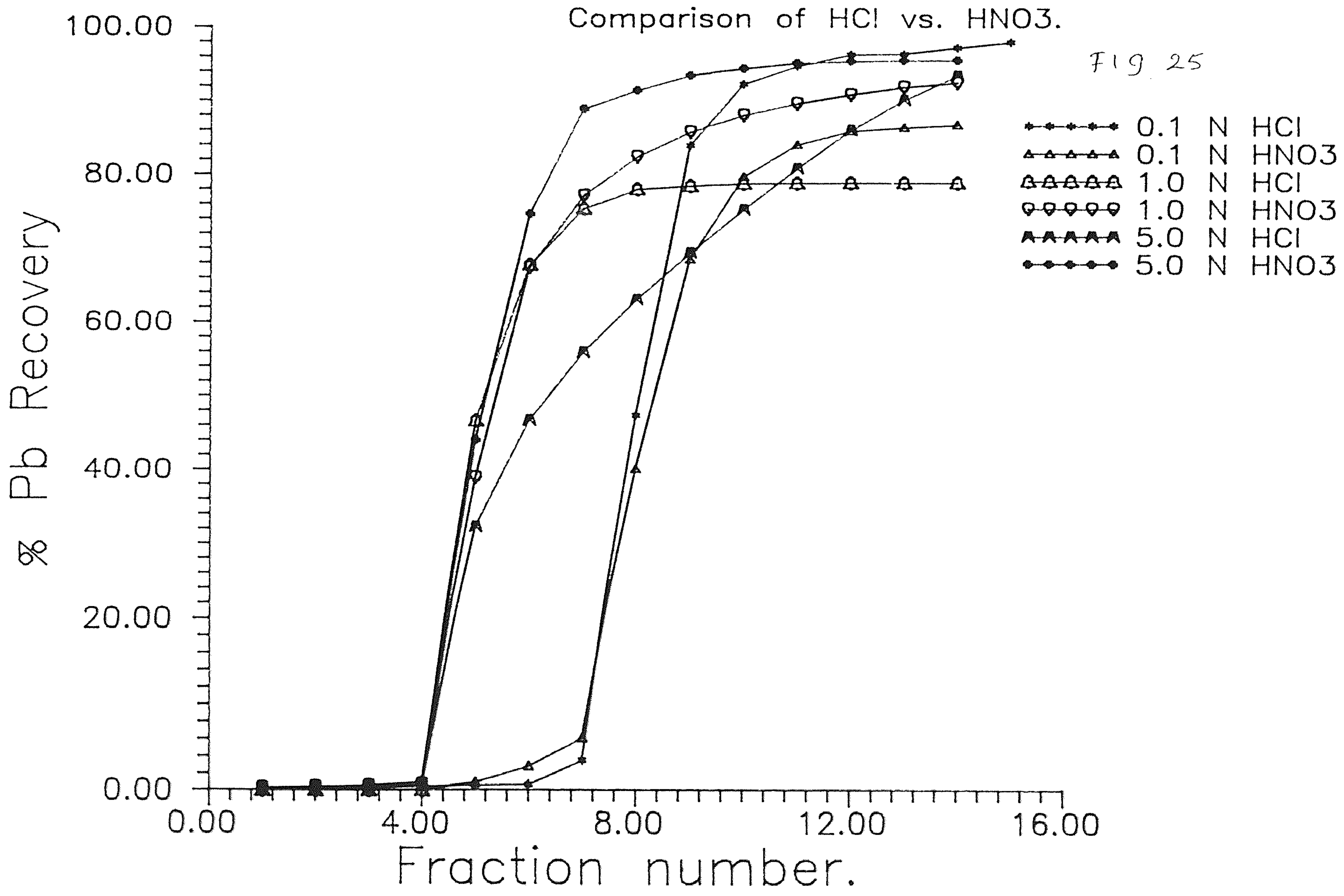

Comparison of HCl vs. HNO3.
% Pb Recovery
100.00
80.00
60.00
40.00
20.00
0.00
Fraction number.
0.00
4.00
8.00
12.00
16.00
0.1 N HCl
0.1 N HNO3
1.0 N HCl
1.0 N HNO3
5.0 N HCl
5.0 N HNO3

FIG 25

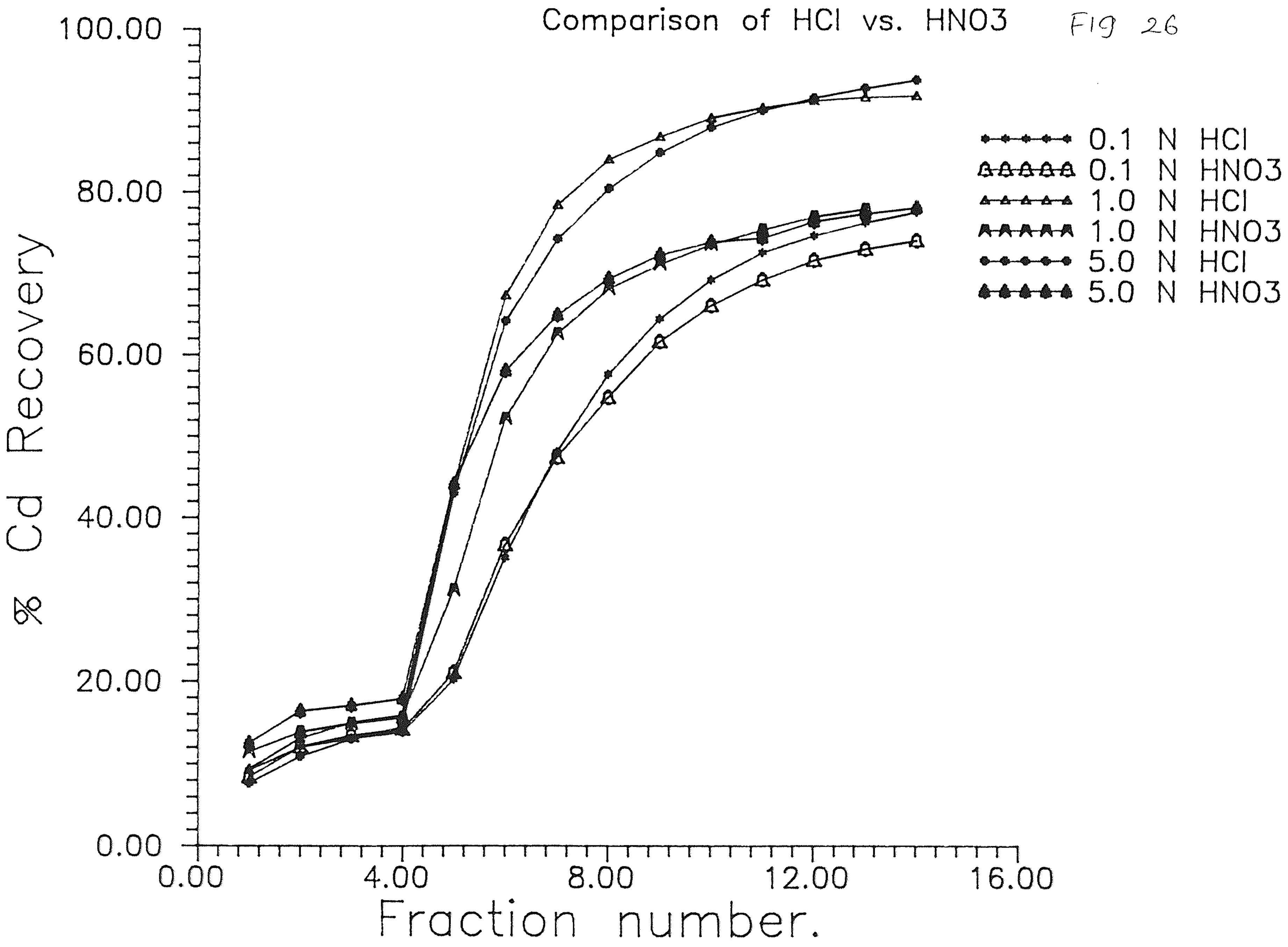

Comparison of HCl vs. HNO3
% Cd Recovery
100.00
80.00
60.00
40.00
20.00
0.00
0.00
4.00
8.00
12.00
16.00
Fraction number.
0.1 N HCl
0.1 N HNO3
1.0 N HCl
1.0 N HNO3
5.0 N HCl
5.0 N HNO3

Fig 26

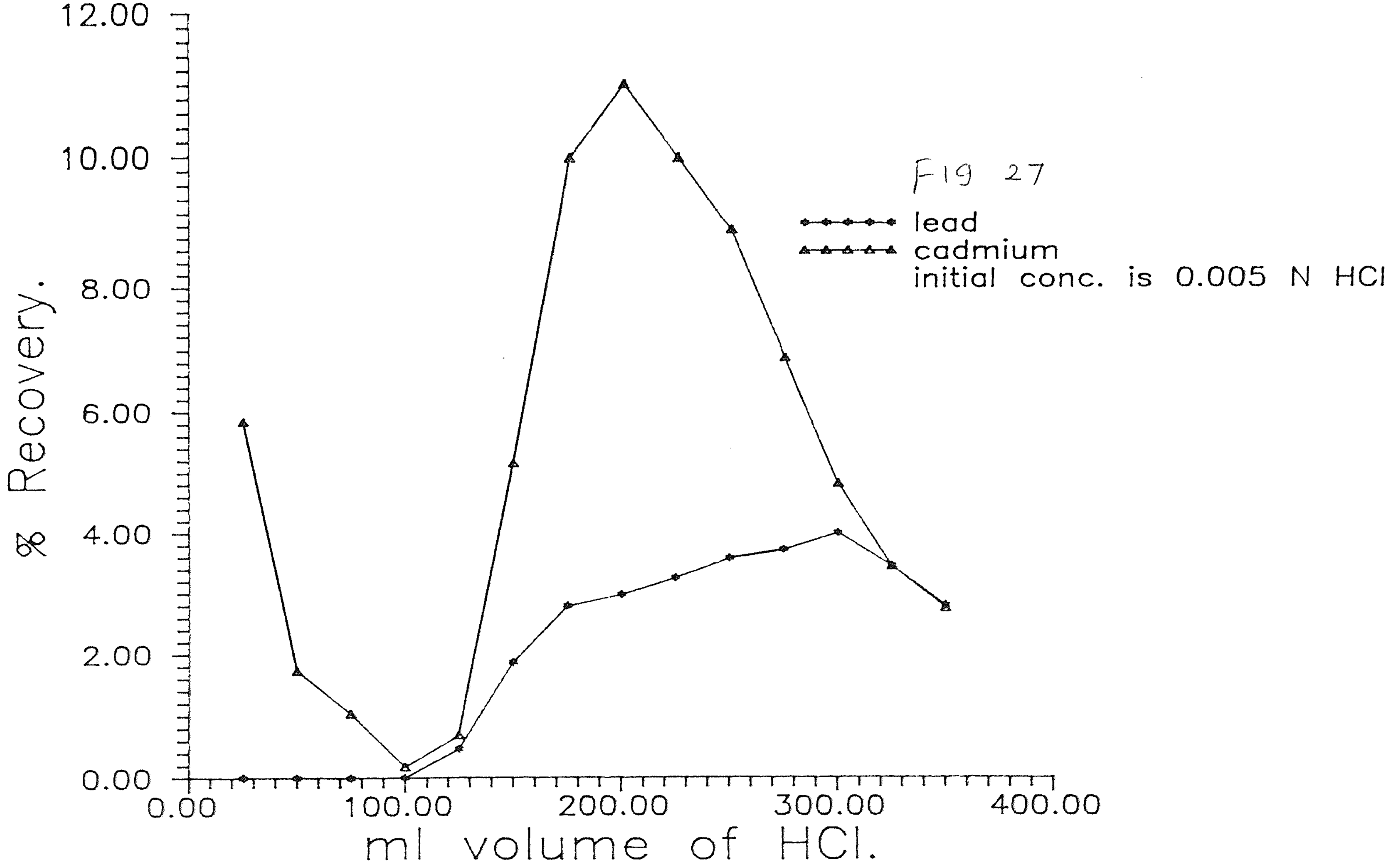
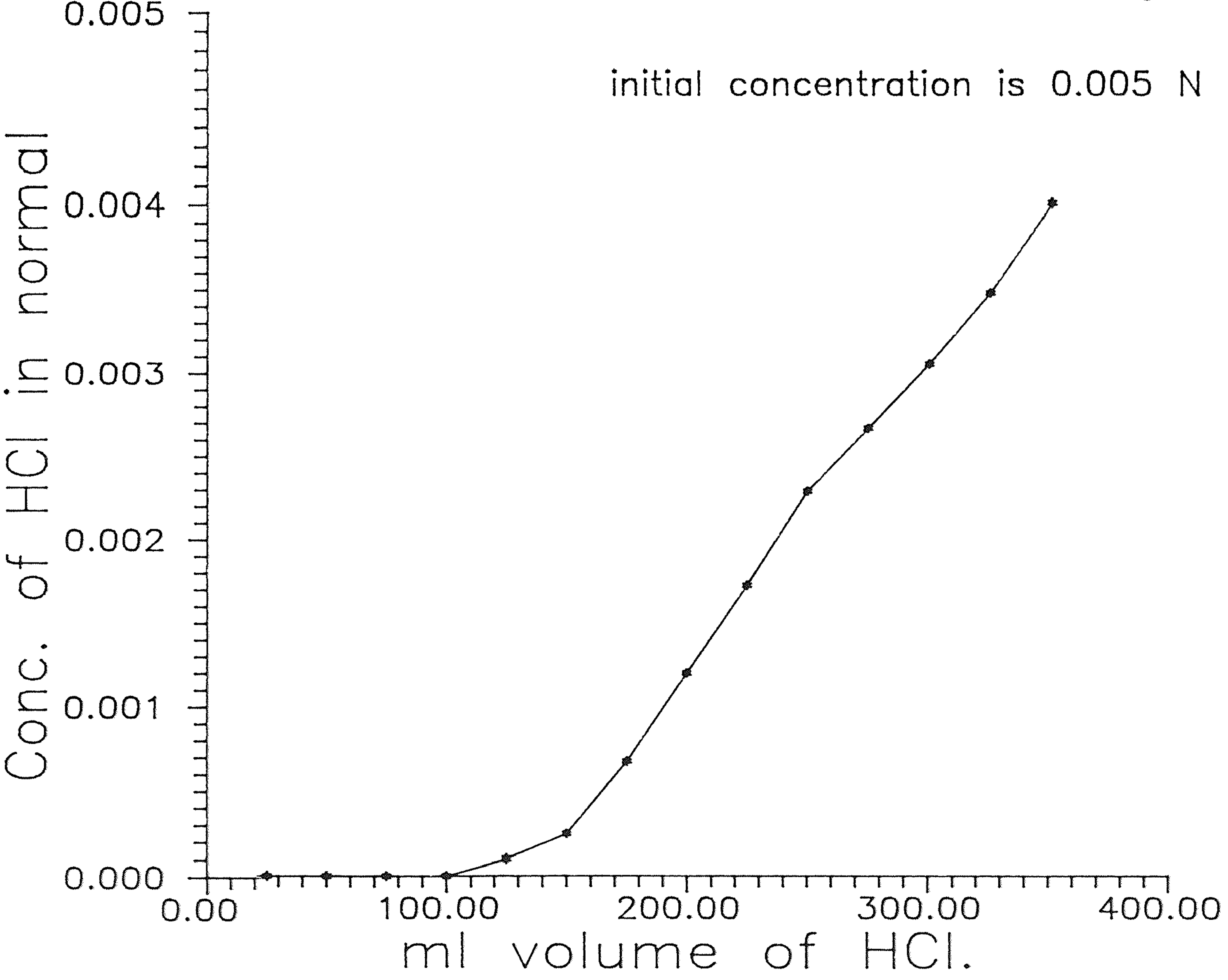


Fig 27
12.00
10.00
8.00
6.00
4.00
2.00
0.00
% Recovery.
0.00
100.00
200.00
300.00
400.00
ml volume of HCl.
lead
cadmium
initial conc. is 0.005 N HCl

Fig 28


initial concentration is 0.005 N HCl.
Conc. of HCl in normal
0.005
0.004
0.003
0.002
0.001
0.000
0.00
100.00
200.00
300.00
400.00
ml volume of HCl.

R-2

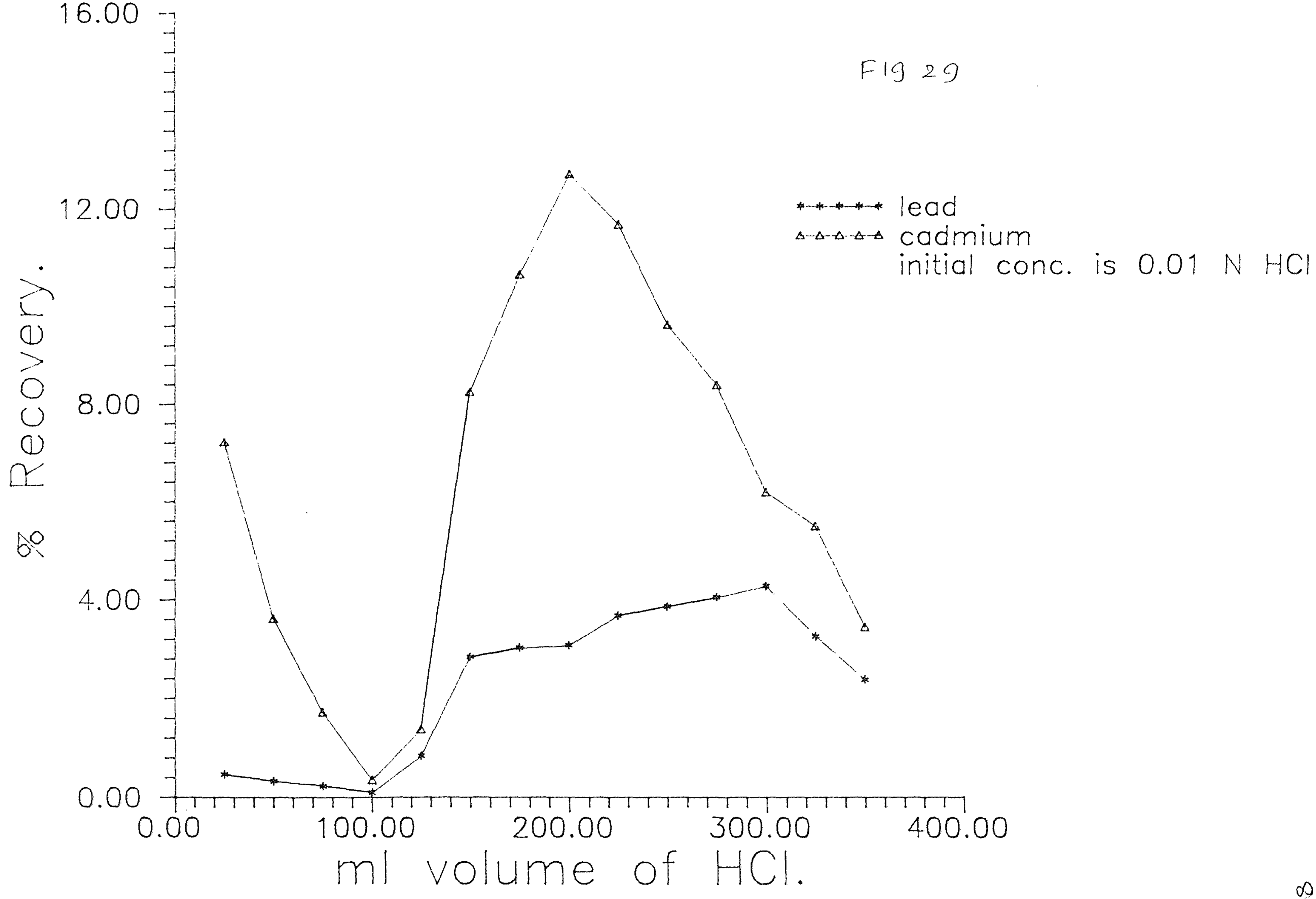


Fig 29
16.00
12.00
8.00
4.00
0.00
% Recovery.
0.00
100.00
200.00
300.00
400.00
ml volume of HCl.
lead
cadmium
initial conc. is 0.01 N HCl

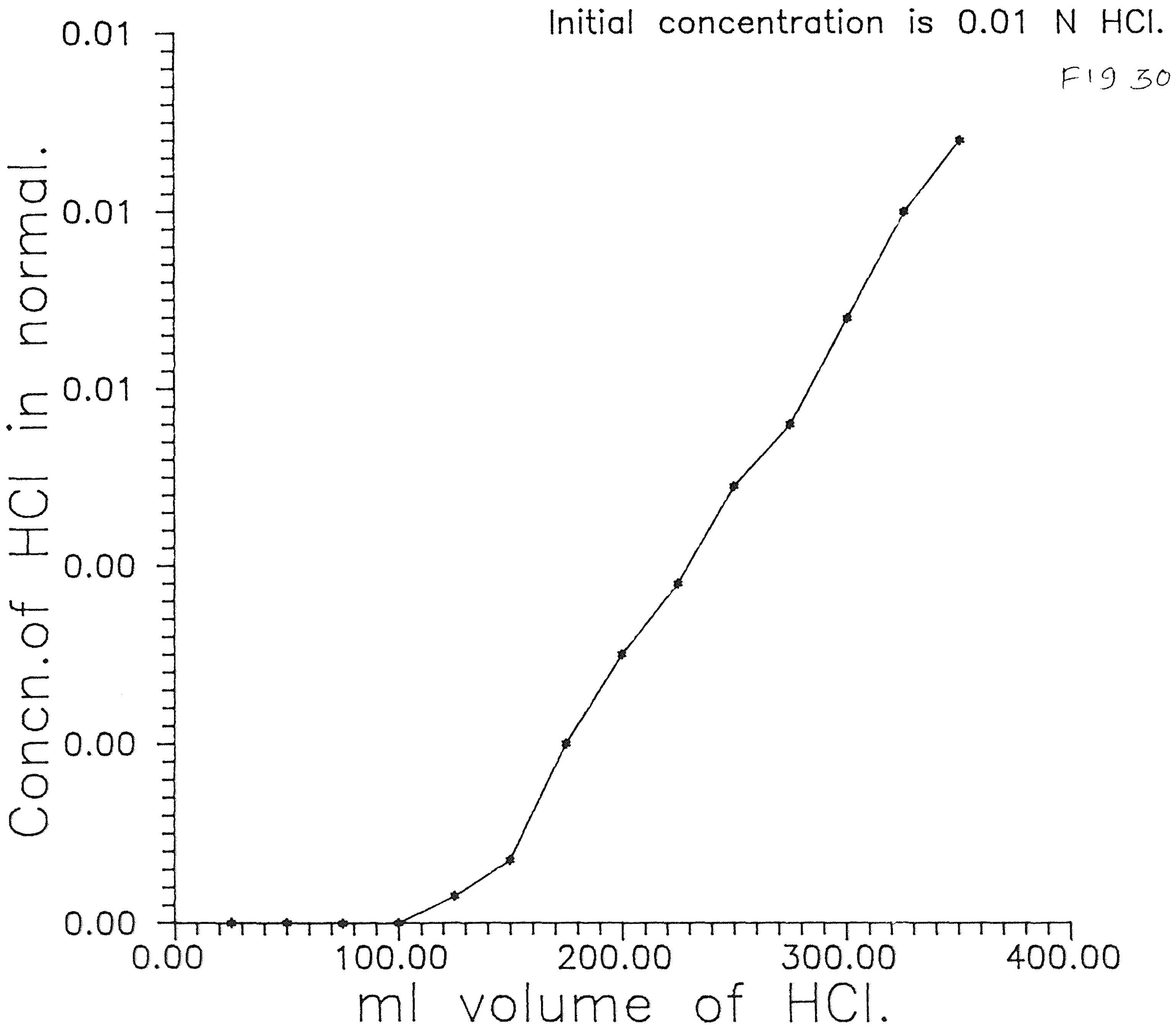

Initial concentration is 0.01 N HCl.
Concn.of HCl in normal.
0.01
0.01
0.01
0.00
0.00
0.00
0.00
100.00
200.00
300.00
400.00
ml volume of HCl.

Fig 30

Thesis N7.
P.2

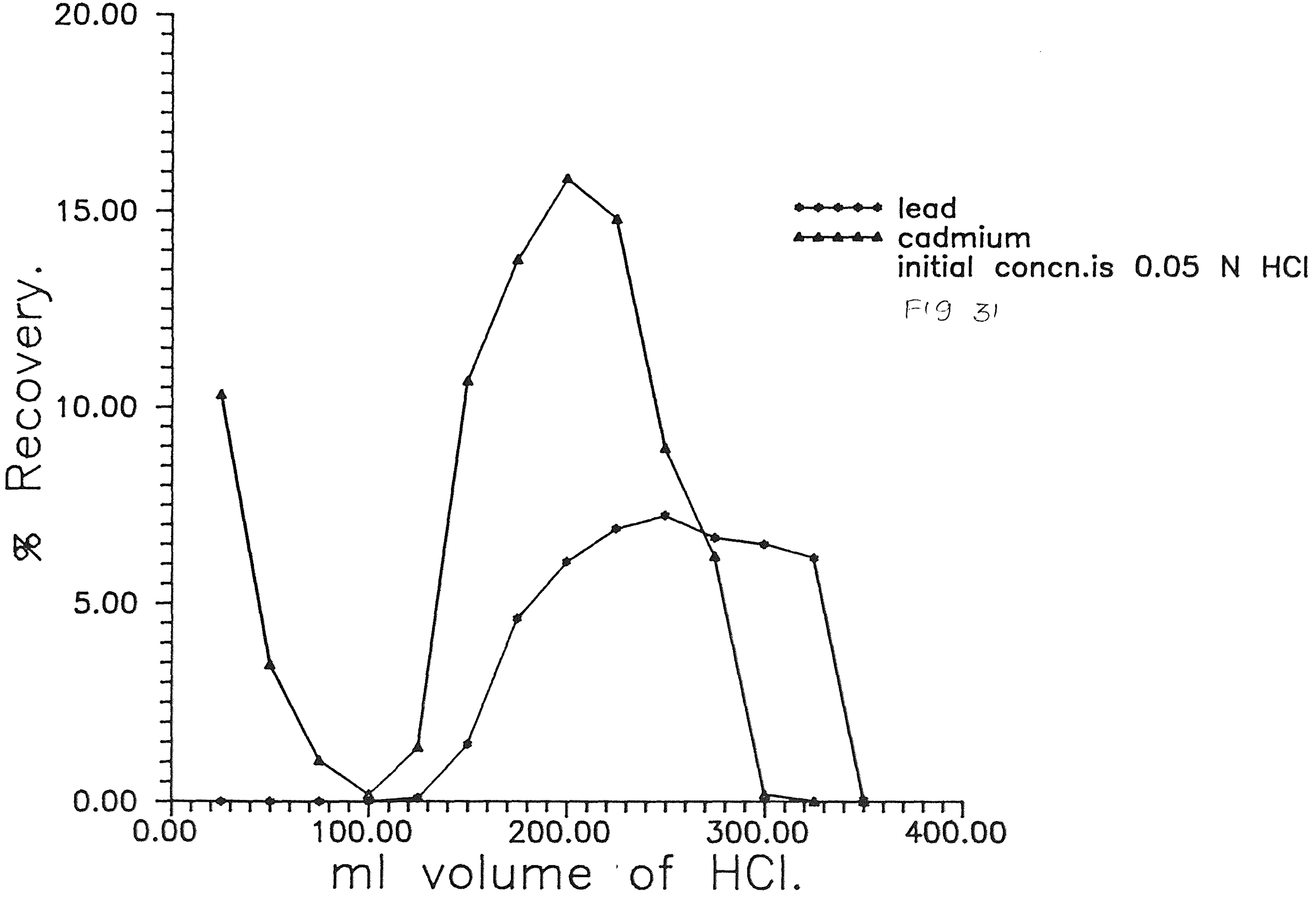
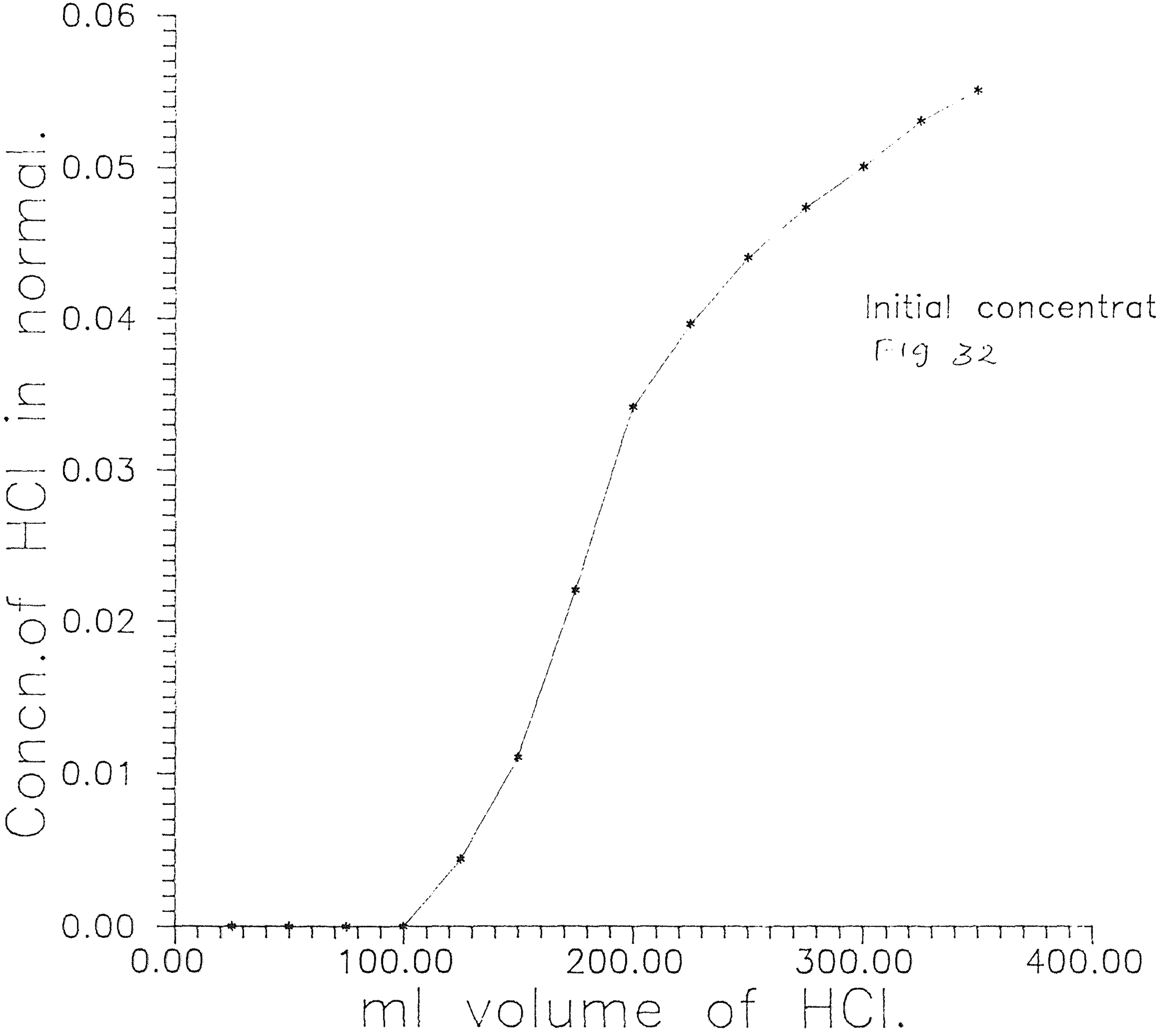
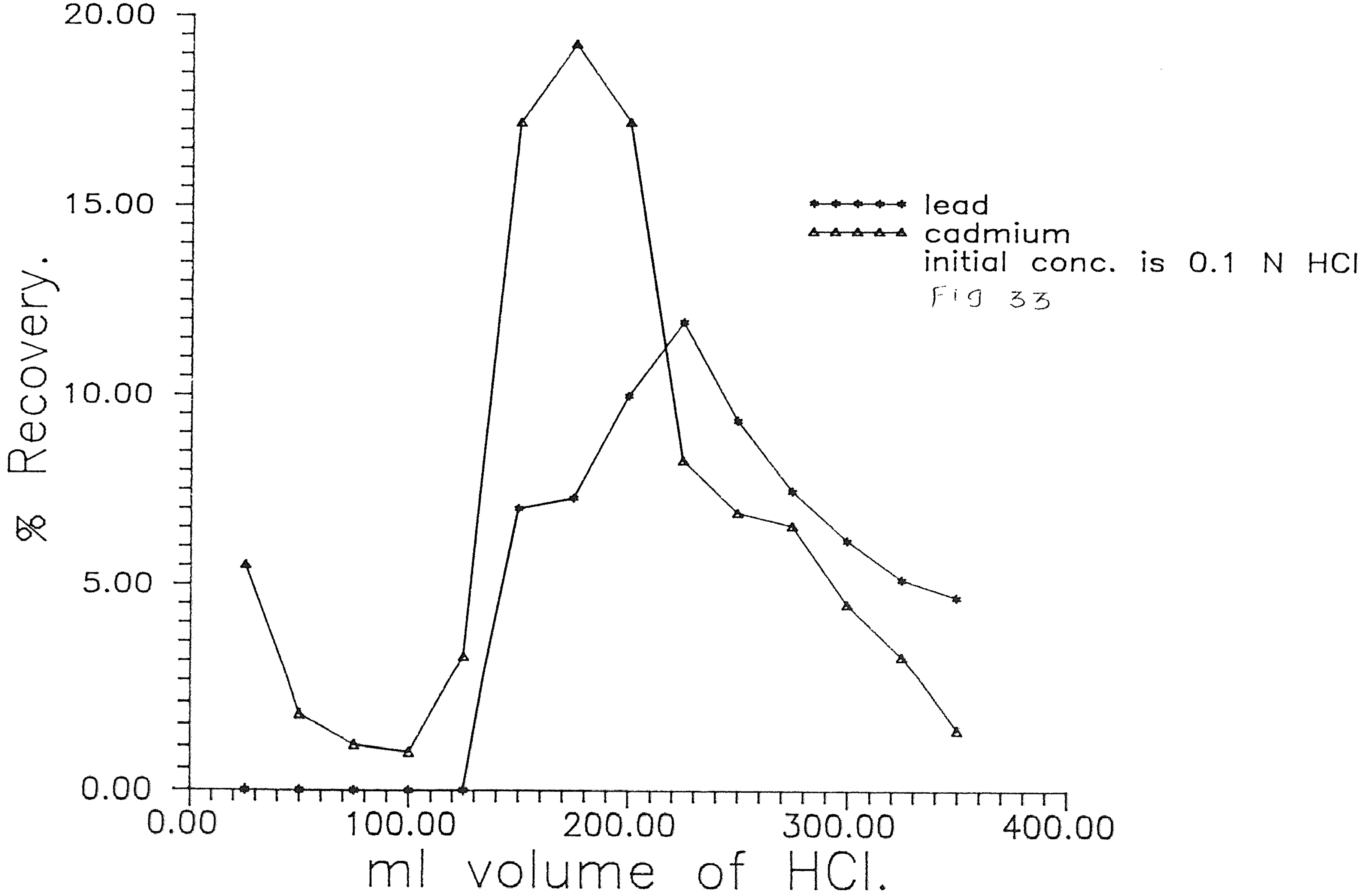


FIG 31



Initial concentration is 0.05 N HCl.

Fig 32


lead
cadmium
initial conc. is 0.1 N HCl
Fig 33
% Recovery.
20.00
15.00
10.00
5.00
0.00
0.00
100.00
200.00
300.00
400.00
ml volume of HCl.

Pbcd4T2

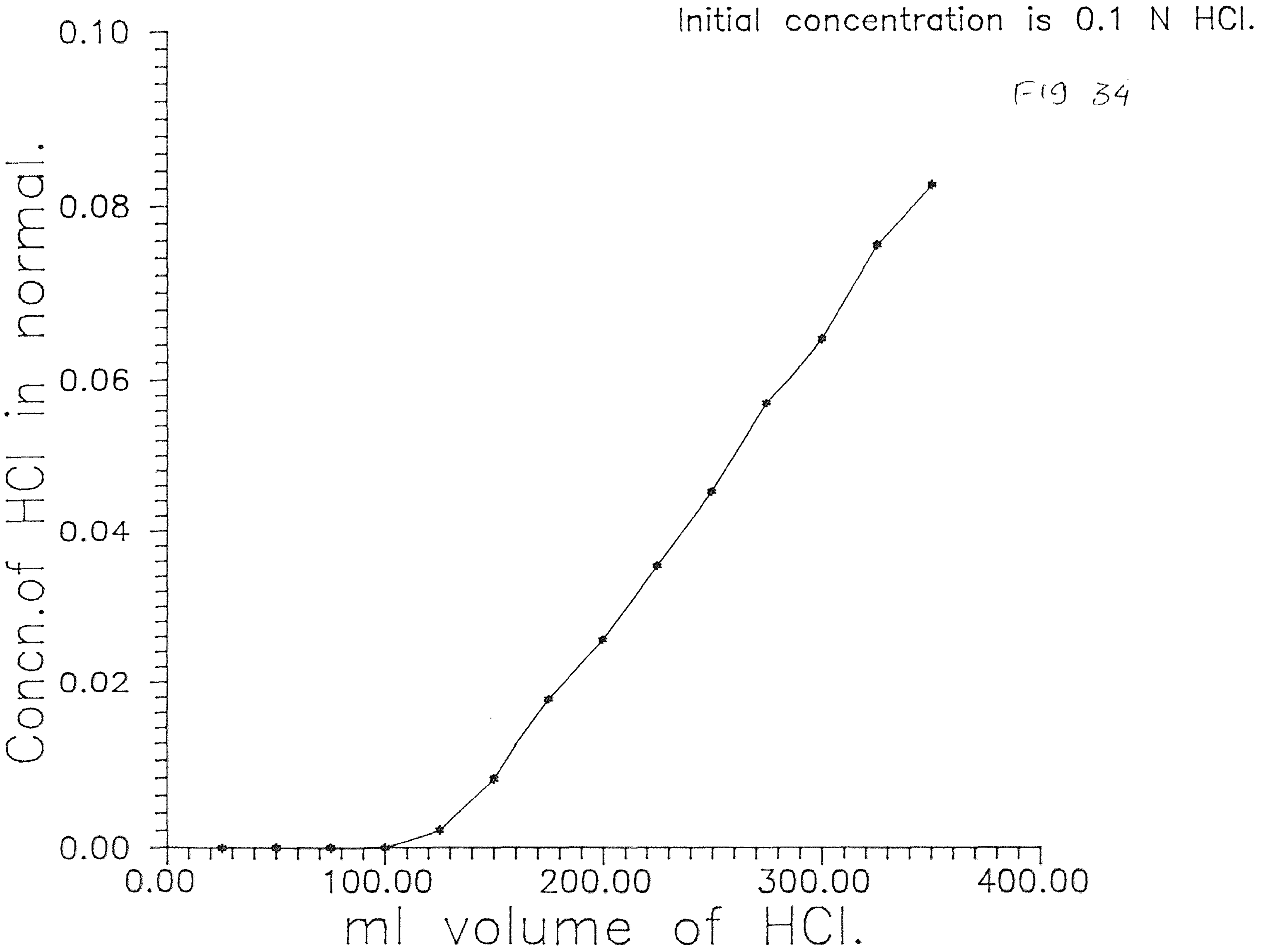


Fig 34