FACULTY PROFILE

1. Name: Dr. Nitin Dattatray Nikam
2. Designation: Assistant Professor
3. Department : Chemistry
4. Address: A/P Rethare Harnax
5. Date of Birth: 06/06/1982
6. Mobile No.: 9767600262
7. E-mail: nikamndchem@gmail.com
8. Qualification: M.Sc. NET, Ph.D.

Sr. No.	Exam/ Degree	Board / University	Subjects / Topics	Year of Passing
1	Ph. D	S U Kolhapur	Chemistry	2017
2	NET	HRDG New Delhi	Chemical Science	2007
3	P.G.	S P P U Pune	Inorganic Chemistry	2005
4	U.G.	S U Kolhapur	Chemistry	2003
5	HSC	Kolhapur	Science	2000
6	SSC	Kolhapur	Mar, Eng, Hin, Sci, Maths, Soc.Sci	1998

9. Teaching Experience: 13 Years

Sr. No.	Institution	Duration	Classes Taught	Total Years
1	S G M College Karad	01/03/2009 to 26/06/2009	B. Sc. I,II,III	0.3
2	Dahiwadi College Dahiwadi	27/06/2009 to 15/07/2009	B. Sc. I,II,III	0.1
3	Abasaheb Marathe College Rajapur	16/07/2009 to 15/06/2016	B. Sc. I,II,III	7
4	D P Bhosale College Koregaon	16/06/2016 to Till	B. Sc. I,II,III M.Sc I, II	5

10. Research Experience: 12 Years

11. Research Guidance:

(PG and Research Guide)

Sr. No.	P.G./M.Phil./ Ph.D.	Year	University / Institute	No. of students awarded	No. of student working

12. Research Projects: 02

Sr. No.	Title	Govt. & Non Govt. Funding Agency	Duration	Grant Received
1	Reverse phase partition Chromatographic Separation studies of some Toxic metal ions	UGC (WRO)Pune	2 Yrs	85000/-
2	Biological specimen preservation by potash alum crystal	D P Bhosale College Koregaon	1 Yr	80,000/-

13. Patents: 01

Sr. No.	Name of the Inventor	Title of Invention with application no.	Date of Filling	Date of Publications	Status
1	Dr P S Patil	Biological specimen preservation by potash alum crystal	22/02/2019	01/03/2019	Published

14. Research Publication**:** (Research papers published in the Journals notified on UGC website)

International Publications (Before 2017 & After 2017)

Sr. No	Title of paper	Name of the authors	Department	Name of Journal	Year of publication	ISSN number	Whether Indexed/ peer reviewed?	Impact factor if any.	Link to the recognition in UGC enlistment of the Journal
1	Column chromatographic separation of uranium (vi) and other elements using Poly(dibenzo-18-crown-6) and glycine medium	Dr P N kamble Dr. N. D Nikam, Dr. P.S. Patil Prof. B.S. Mohite	Chemistry	Proceedings of the fourteenth biennial DAE-BRNS symposium on nuclear and radiochemistry	2019	--	--	--	
2	Sorption study of beryllium (II) in glycine medium using poly [dibenzo-18 crown-6] and column chromatography	Dr. N. D Nikam, Dr. P.S. Patil Prof. B.S. Mohite	Chemistry	International Journal of Chemical Science	**2018**	2523-6075	Peer reviewed	5.22	
3	Sorption Study of Bismuth (III) in Glycine Medium Using Poly[dibenzo-18 crown-6] and Column Chromatography	Dr. N. D Nikam, Dr. P.S. Patil Prof. B.S. Mohite	Chemistry	International Journal of Research and Analytical Reviews	**2018**	2349-5138	Peer reviewed	4.236	
4	Sorption study of Cadmium(II) in glycine medium using poly [dibenzo-18 crown-6] and	Dr. N. D Nikam, Dr. P.S. Patil Prof.	Chemistry	International Journal of Chemistry Studies	**2018**	2581-348X	Peer reviewed	5.44	

	Column chromatography	B.S. Mohite							
5	Sorption Study of Chromium(III) in Glycine Medium Using Poly [dibenzo-18 crown-6] and Column Chromatography	Dr. N. D Nikam, Dr. P.S. Patil Prof. B.S. Mohite	Chemistry	Journal of Applicable Chemistry	**2018**	2278-1862	Peer reviewed	1.211	
6	Sorption Study of Nickel(II) in Glycine Medium Using Poly[dibenzo-18 crown-6] and Column Chromatography	Dr. N. D Nikam, Dr. P.S. Patil Prof. B.S. Mohite	Chemistry	Journal of Applicable Chemistry	**2018**	2278-1862	Peer reviewed	1.211	
7	A sorption and separation study of strontium (II) using poly [dibenzo-18-crown-6] in picric acid.	Dr. N. D Nikam, Dr. P.S. Patil Prof. B.S. Mohite	Chemistry	International Journal of Chemical Science and Technology	**2016**	2249-8532	Peer reviewed	--	

National Publications (Before 2017 & After 2017)									
Sr. No	Title of paper	Name of the authors	Department	Name of Journal	Year of publication	ISSN number	Whether Indexed/ peer reviewed?	Impact factor if any.	Link to the recognition in UGC enlistment of the Journal

15. Research Citations:

Sr. No.	Source	Citation	H- Index	i-10 Index
1	Scopus			
2	Publons/ Web of Science			
3	Google Scholar			
4	Research Gate			

5	ORICID			
6	Vidwan			
7	LinkedIn			

16. Books, Book chapters and papers published in conference proceedings :

International Conferences proceedings /webinar/ Workshops/ Seminars (Before 2017 & After 2017)								
Sr. No	Title of the book/ chapters published	Title of the paper	Title of the proceedings /Poster/ Oral Presentation	Name of the conference	Year of publication	ISBN/ ISSN number of the proceeding	Affiliating Institute at the time of publication	Name of the publisher

National Conferences proceedings /webinar / Workshops/ Seminars (Before 2017 & After 2017)								
Sr. No	Title of the book/ chapters published	Title of the paper	Title of the proceedings /Poster/ Oral Presentation	Name of the conference	Year of publication	ISBN/ ISSN number of the proceeding	Affiliating Institute at the time of publication	Name of the publisher

17. Participation in International / National conferences, Webinar, Seminars, Workshops:

Sr. No.	Department	Name of conference/ Webinar, Seminars and Workshop	Level International/National/State/University	Date	Place
1	Chemistry	Revised syllabus in chemistry at B. Sc I	University	11/8/2018	Kolhapur
2	Chemistry	National Level Online Quize on Grneral Chemistry	National	13/08/2020	Nagpur

18. Resource Person/Chairperson in National/International/Sate/University Level conferences, Webinars, Seminars, Workshops:

Sr. No.	Department	Name of conference, Webinar, Seminars and Workshop	Role Resource Person/ Chairperson	Level International/ National/ State/ University	Date	Place
1						

19. Awards / Fellowship:

Sr. No.	Level (International, National, State, University, Other)	Awards /Fellowship	Awarding Agency / Institute	Year / Duration

20. Participation in Academic Events: Orientation /Refresher Courses/FDP/MOOCs/Swayam courses etc.

Sr. No.	Organizing Institute	Event	Duration	Year	Grade Achieved
1	UGC HRDC, Pune	Orientation Programme	03-08-2015 to 30-08-2015	2015	A
2	UGC HRDC, Aurangabad	Refresher Course	03-03-2018 to 22-03-2018	2018	A
3	SWAYAM, ARPIT	Refresher Course	January to February,2020	2020	B
4	TCS, Pune	Faculty Development Programme	26-09-2016 to 01-10-2016	2016	--
5	Infosys, Mysore	Faculty Development Programme	22-08-2016 to 30-08-2016	2016	--
6	BOSCH, Banglore	TTT, Programme	04-09-2018 to 08-09-2018	2018	--
7	A.U., College, Heyderabad	Faculty Development Programme	27-01-2021 to 02-02-2021	2021	--
8	NPTEL, HRD, India	Short Term Course	August – September 2019	2019	--

21. Organizational Experience:

Sr. No.	Event/program	Title	Level	Role	Date
1	Workshop				
2	Seminar				
3	Conference	New Horizons in Green Chemistry and Toxicology	National	Organizing Secretary	2/04/2019
		“HYPHENATED TECHNIQUES IN DETECTION OF SMART MATERIALS” (IWHTDSM)	International	Organizing Committee	11/07/2020
4	Avishkar				
5	Webinar				
6	Other				

22. Any other information

महाराष्ट्र राज्य माध्यमिक व उच्च माध्यमिक शिक्षण मंडळ, पुणे.

Maharashtra State Board Of Secondary and Higher Secondary Education, Pune.

माध्यमिक शालान्त प्रमाणपत्र परीक्षा

SECONDARY SCHOOL CERTIFICATE EXAMINATION

असे प्रमाणित करण्यात येते की,
This is to certify that NIKAM NITIN DATTATRYA

विभागीय मंडळ Divisional Board	आसन क्रमांक Seat No.	केंद्र क्रमांक Centre No.	शाळा क्रमांक School No.	प्रमाणपत्राचा अनुक्रमांक Sr. No. of Certificate
KOLHAPUR	F068437	1027	22.150	031619

माध्यमिक शालान्त प्रमाणपत्र परीक्षा
has passed the SECONDARY SCHOOL CERTIFICATE EXAMINATION MARCH-1998
श्रेणीमध्ये खाली दर्शविलेले विषय घेऊन उत्तीर्ण झाला/झाली आहे.
in Grade FIRST with subjects shown below

मुख्य विषय Main Subjects	कमाल गुण Max. Marks	प्राप्त गुण Marks Obtained	श्रेणीचे विषय Subjects of Grade	श्रेणी Grade
MARATHI	100	62	[WORK EXPERIENCE/TECH]	
HINDI	100	60	PREPARATION STATIONARY [SCHOOL SUBJECTS]	A
ENGLISH	100	50	HEALTH & PHYSICAL EDU.	A
MATHEMATICS	150	99	SOCIAL SERVICE	A
SCIENCE	150	127	VALUE EDUCATION	A
SOCIAL SCIENCES	150	94	GENERAL KNOWLEDGE	A
एकूण गुण Total Marks	750	492	PERCENTAGE =====>	65.60

एकूण प्राप्त गुण (अक्षरीं)
Total Marks Obtained (in Words) * FOUR HUNDRED AND NINETYTWO *

जन्म दिनांक
Date of Birth 06-06-1982 (SIXTH JUNE NINETEEN HUNDRED EIGHTY TWO)

उमेदवाराने माझ्या समक्ष पुढील चौकटीत स्वाक्षरी केली आहे.
The candidate has signed before me in the adjoining frame

KOLHAPUR
29th June 1998

शाळाप्रमुखाची स्वाक्षरी व शाळेचा शिक्का
Signature of the Head of the School with School Stamp.

HEAD-MASTER,
Pathe Bapurao Vidyalaya.
Rethare Harnax, (Sangli)

Y 0228589

विभागीय सचिव
Divisional Secretary

महाराष्ट्र राज्य माध्यमिक व उच्च माध्यमिक शिक्षण मंडळ, पुणे.
Maharashtra State Board of Secondary and Higher Secondary Education, Pune.

KOLHAPUR **DIVISIONAL BOARD** विभागीय मंडळ

उच्च माध्यमिक प्रमाणपत्र परीक्षेचे गुणपत्रक
STATEMENT OF MARKS OF THE HIGHER SECONDARY CERTIFICATE EXAMINATION

केंद्र क्रमांक CENTRE NO.	जिल्हा व उच्च माध्यमिक शाळा क्रमांक DIST. & HR. SEC. SCHOOL NO.	परीक्षेचा आसन क्रमांक EXAMINATION SEAT NO.	उमेदवाराचे संपूर्ण नाव (आडनाव प्रथम) CANDIDATE'S FULL NAME (SURNAME FIRST)	परीक्षेचा महिना आणि वर्ष MONTH AND YEAR OF EXAMINATION	शाखा STREAM	गुणपत्रिकेचा अनुक्रमांक SERIAL NO. OF STATEMENT
177	22. 022	I011140	NIKAM NITIN DATTATRYA	FEB-2000	SCIENCE	032397

परीक्षेस घेतलेले विषय SUBJECTS OFFERED	अनिवार्य भाषा COMPULSORY LANGUAGES		वैकल्पिक विषय OPTIONAL SUBJECTS					व्यावसायिक विषय VOCATIONAL SUBJECT		गुणांची टक्केवारी PERCENTAGE OF MARKS
* विषयाचा सांकेतिक क्रमांक * SUBJECT CODE ⇒	01	02	39	40	54	55	--	--	एकूण गुण TOTAL MARKS	46.00
कमाल गुण MAXIMUM MARKS	100	100	100	100	100	100	100	200	600/700	निकाल RESULT
प्राप्त गुण ⇒ MARKS OBTAINED	045	055	066	035	035	040	--	--	276	PASS

एकूण गुण (अक्षरी) TOTAL MARKS (IN WORDS) * TWO HUNDRED AND SEVENTYSIX *

विभागीय सचिव
DIVISIONAL SECRETARY
MSBSHSE

प्रमाणपत्राच्या श्रेणी / Grades of Certificates

विशेष प्राविण्यासह प्रथम श्रेणी (Grade I with Distinction)	प्रथम श्रेणी (Grade I)	द्वितीय श्रेणी (Grade II)	उत्तीर्ण श्रेणी (Grade Pass)
७५ % व त्यापेक्षा अधिक गुण 75% and above	६०% व त्यापेक्षा अधिक परंतु ७५% पेक्षा कमी गुण 60% and above but below 75%	४५% व त्यापेक्षा अधिक परंतु ६०% पेक्षा कमी गुण 45% and above but below 60%	इतर सर्व उत्तीर्ण उमेदवार (विषयात सूट घेतलेल्या उमेदवारासह) All other successful candidates (Including the exempted)

महत्त्वाचे - या गुणपत्रिकेत कोणताही बदल करता येणार नाही. असा बदल केल्यास ही गुणपत्रिका रद्द होईल, व मंडळाच्या निर्णयानुसार योग्य ती इतर दंडात्मक कारवाई होईल.

IMPORTANT - NO CHANGE in this Statement of Marks shall be made. Any infringement in the Statement will result in its cancellation and will involve imposition of other appropriate penalty as decided by the Board.

* सांकेतिक क्रमांकानुसार विषय मागील पृष्ठावर पहावेत.
* See the subject codes overleaf.

टीप/Note
XX - ही खूण विषयात सूट दर्शविते. XX - Indicates Exemption in the subject.
AA - ही खूण अनुपस्थिती दर्शविते. AA - Indicates Absent.
उत्तीर्ण होण्यासाठी प्रत्येक विषयात किमान ३५ % गुण आवश्यक.
To pass minimum 35% marks are essential in each subject.
एकूण गुणात + ही खूण असल्यास ती मंडळ नियमानुसार प्रथम श्रेणीसाठी आवश्यक जादा गुण दर्शविते.
+ Sign, if shown under the total marks, indicates the grace marks awarded for getting grade I, as per the regulation of the Board.

V 0067039

महाराष्ट्र राज्य माध्यमिक व उच्च माध्यमिक शिक्षण मंडळ, पुणे

Maharashtra State Board Of Secondary and Higher Secondary Education, Pune

उच्च माध्यमिक शालान्त परीक्षा प्रमाणपत्र

HIGHER SECONDARY SCHOOL EXAMINATION CERTIFICATE

असे प्रमाणित करण्यात येते की / This is to certify that

NIKAM NITIN DATTATRYA

विभागीय मंडळ Divisional Board	आसन क्रमांक Seat No.	केन्द्र क्रमांक Centre No.	उच्च माध्यमिक शाळा क्रमांक Higher Secondary School No.	प्रमाणपत्राचा अनुक्रमांक Sr. No. of Certificate
KOLHAPUR	I011140	177	22.022	019637

उच्च माध्यमिक शालान्त प्रमाणपत्र परीक्षा (१० + २ आकृतीबंधानुसार)
has passed the HIGHER SECONDARY SCHOOL CERTIFICATE EXAMINATION FEBRUARY-2000
(Under 10 + 2 Pattern)

in Grade II श्रेणीमध्ये खाली दर्शविलेले विषय घेऊन उत्तीर्ण झाला/झाली आहे.
with subjects shown below.

सांकेतिक क्रमांक व विषय Subject Code No. and Subjects	कमाल गुण Max. Marks	प्राप्त गुण / Marks Obtained In Figures	 In Words
01 ENGLISH	100	045	FORTY FIVE
02 MARATHI	100	055	FIFTY FIVE
39 GEOGRAPHY	100	066	SIXTY SIX
40 MATHS & STATS.	100	035	THIRTY FIVE
54 PHYSICS	100	035	THIRTY FIVE
55 CHEMISTRY	100	040	FORTY
एकूण गुण /Total Marks Percentage 46.00	600	276	TWO HUNDRED AND SEVENTY SIX

1119649
KOLHAPUR
25TH MAY 2000

023542165

विभागीय सचिव / Divisional Secretary

SHIVAJI UNIVERSITY KOLHAPUR

PRN. : BS01003493
Seat No. : 16352

Sr. No. : 2002/ 209355

STATEMENT SHOWING THE NUMBER OF MARKS OBTAINED BY

Shri/Smt. NIKAM NITIN DATTATARAY
in each head of passing at the B.SC.PART - III

COLLEGE : PALUS
Examination Apr. 2003

SUBJECT NAME	Theory Max	Theory Min	Theory Obt	Practical Max	Practical Min	Practical Obt
1. ENGLISH	100	35	53	---	---	---
2. Chemistry Paper V	100	35	81	---	---	---
3. Chemistry Paper VI	100	35	69	---	---	---
4. Chemistry Paper VII	100	35	87	---	---	---
5. Chemistry Paper VIII	100	35	64	---	---	---
6. Chemistry Practical	---	---	---	200	70	166

Total Of Part-1(Excluding ENG) 409/1000
Total Of Part-2 521/900
Total Of Part-3(Excluding ENG) 467
Grand Total (Out Of 2500) 1397

Total Marks Obtained (in Words) : ONE THOUSAND THREE HUNDRED AND NINETY SEVEN OUT OF TWO THOUSAND FIVE HUNDRED

Result : SECOND CLASS 55.88%

Kolhapur, Date : 16/5/2003

CONTROLLER OF EXAMINATIONS

UNIVERSITY OF PUNE

GANESHKHIND, PUNE 411 007.

M. Sc. INORGANIC CHEMISTRY

NO.: 04 - 0215679

STATEMENT OF MARKS FOR
APR/MAY 2005

SEAT NO. 32079 CENTRE 16 PERM. REG. NO.: IN0332042
NAME NIKAM NITIN DATTATRAY
COLLEGE 032

YEAR	COURSE NAME	MARKS OBTAINED TEX UEX TOT
CH-107	PHYSICAL CHEMISTRY PRACTICALS	15 068 083
CH-110	PHYSICAL CHEMISTRY I	15 038 053
CH-127	INORGANIC CHEMISTRY PRACTICALS	17 065 082
CH-130	INORGANIC CHEMISTRY I	13 043 056
CH-150	ORG.REACTION MECHA.&STEREOCHEM	12 046 058
CH-210	PHYSICAL CHEMISTRY II	17 041 058
CH-230	INORGANIC CHEMISTRY II	11 056 067
CH-247	ORGANIC CHEMISTRY PRACTICALS	16 069 085
CH-250	SYNTHETIC ORG.CHEM& SPECTROSCO	18 047 065
CH-290	GENERAL CHEMISTRY	18 038 056
CH-326	ORGANOMETA.COMP.IN SYN/HOM CAT	16 040 056
CH-330	COORDI.COMP.& STRUCTURAL MTD.	13 037 050
CH-331	INORGANIC REACTION MECHANISM	11 047 058
CH-332	METALLOPROTEINS & BIOINOR.MED.	13 033 046
*CH-387	QUANTI.INORG.ANA. (PRACT.)	15 068 083
*CH-388AB	PRACT.ON SYN/STR MTD/COORD.COM	16 064 080
*CH-430	INO.SOLIDS,HETERO.CAT;STRU.MTD	15 049 064
*CH-431	MATERIAL & INDUSTRIAL INO.CHEM	11 040 051
*CH-445	INO.APPLI.IN MATE.SC/BIOTE/ENV	13 056 069
*CH-488	RESEARCH PROJECT	14 062 076

University Total : 990 Departmental Total : 306 Grand Total : 1296

Result : FIRST CLASS

CONTROLLER OF EXAMINATIONS

STATEMENT NO.: 39 DATE: 19/07/2005

#=0.4 & =0.163 $=0.1 @=0.1&0.163 *=CURRENT APPEARANCE

AA-ABSENT EE-EXTERNAL FFF-FAIL IN COURSE TEX-TERM END EXAMINATION UEX-UNIVERSITY EXAMINATION PP-PRACTICAL EXAMINATION

REGISTERED

वैज्ञानिक तथा औद्योगिक अनुसंधान परिषद्
मानव संसाधन विकास समूह, परीक्षा एकक
COUNCIL OF SCIENTIFIC & INDUSTRIAL RESEARCH
Human Resource Development Group, Examination Unit
सी.एस.आई.आर. कॉम्पलेक्स, लाइब्रेरी एवेन्यू, पूसा, नई दिल्ली-110012
CSIR Complex, Library Avenue, Pusa, New Delhi-110012

F.No.10-2(5)/2006(ii)-E.U.II

Dated: 01/06/2007

ROLL NO. : 102981
SH. NIKAM NITIN DATTATRAY
S/O NIKAM DATTATRAY KSHABA
SHOP NO 4-2-357 MOTISINGH GALI
BADI CHAWDI SULTAN BAZAR
HYDERABAD
500095

Sub: Joint CSIR-UGC National Eligibility Test (NET) for Lectureship held on 24.12.2006.

Sir/Madam,

We are happy to inform you that you have qualified in the above examination for consideration for award of ***LECTURESHIP (NET***) in subject ***CHEMICAL SCIENCES*** under the ***"LECTURESHIP - CATEGORY"*** only. subject to the condition that you fulfill all the eligibility criteria as contained in the Examination Notification No.10-2(5)/2006(ii)-E.U.II published in the Employment News issue dated **02nd– 08th September, 2006**. Please ensure that you fulfill all the eligibility conditions.

If you had applied under ***"Result Awaited"*** **category, please note that you have to attain the requisite eligibility criteria latest by 30.04.2008 failing which your candidature will be deemed cancelled.**

You will be required to produce copies of your certificates of proof of date of birth, M.Sc. final year marks sheet (55% marks in case of General/OBC and 50% marks for SC/ST candidates),Caste/Category certificates (if applicable), and M.Sc. or equivalent Degree Certificate duly attested alongwith other relevant papers to the Lectureship appointing authorities.

Since this Test for Eligibility for ***LECTURESHIP (NET)*** was conducted on behalf of University Grants Commission (UGC) this letter may be preserved as a certificate and no separate certificate will be issued by UGC. You are advised to contact the UGC at the following address for any clarification:

The Education Officer (NET) UGC,
University Grants Commission,
Delhi University, South campus,
Benito Juarez Marg, Dhaula Kuan,
New Delhi-110021.

As per rules of the UGC, you are eligible for ***LECTURESHIP (NET)***, if you apply against such a vacancy as may be notified by UGC / an Indian University /College. Though you have not qualified for JRF NET, you are eligible to join as JRF in research scheme projects if otherwise suitable as per the eligibility criteria of research schemes.

This letter may be preserved and treated as a certificate.

Your dossier has already been forwarded to the UGC, Delhi University, South Campus, Benito Juarez Marg, New Delhi-110021 for information and necessary action.

In the event of your having being found ineligible at any stage in future for the award/eligibility for LECTURESHIP due to any reason (including unintentional computer error or printer's devil etc.),this letter will be deemed withdrawn.

Kindly acknowledge the receipt.

Yours faithfully,

(G.K.BHATNAGAR)
CONTROLLER OF EXAMINATIONS

Phone : (011) 25721759, 25848155 • Fax : (91-11) 25840887, 25848155 • E-mail : examunit@csirhrdg.res.in

561026400068

Shivaji University, Kolhapur

WE the Chancellor, Vice-Chancellor *and* Members *of the* Management Council *on the recommendation of the* Academic Council *certify that*

Nikam Nitin Dattatray
Mother's Name - Shailaja

has passed the

Doctor of Philosophy

in Chemistry
in faculty of Science And Technology
on 05 th August, 2017.
The said degree has been conferred on him
at Kolhapur in the Winter Session
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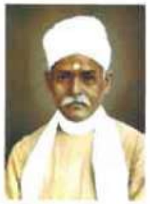

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Original Article

A sorption and separation study of strontium (II) using poly [dibenzo-18-crown-6] in picric acid.

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Abstract

A simple chromatographic separation method has been developed for quantitative sorption of strontium(II) from an aqueous solution of 5×10^{-4} M picric acid using poly [dibenzo-18-crown-6] as stationary phase. The sorption of strontium(II) was quantitative 1×10^{-4} M to 5×10^{-2} M picric acid. The elution of strontium(II) was quantitative with 0.5–6.0 M HCl, 1M HBr, M 1.0M H_2SO_4. The capacity of poly [dibenzo-18-crown-6] for strontium (II) was found to be 0.236 ± 0.01 mmol/g of crown polymer. The effects of concentrations of picric acid, strontium (II), foreign ions and eluents have been studied. strontium(II) was separated from a number of cations in multicomponent mixtures. The applicability of the proposed method was ascertained for the determination of strontium (II) in real samples. The reliability of method was checked by comparison of the results with those obtained using flame photometer. The method is very simple, rapid and selective with good reproducibility (approximately±2%).

Key words:- A sorption and separation, strontium (II), picric acid, poly [dibenzo-18- crown-6].

1.1 Introduction:-

In growing field of separation science, the most challenging research area of present day is due to the growing concern for environment protection and radioactive waste management. Therefore there is an analytical requirement for the trace determination of Strontium (II) in various types of samples. The radioactive isotopes of strontium do not occur naturally but are produced via nuclear fissions.^{90}Sr is main and most hazardous radioactive isotopes of the strontium and other radioactive strontium isotopes have been employed for medical uses. Strontium is considered one of the environmentally hazardous constituents and therefore tends to concentrate in the teeth, bones and blood-forming tissue [1–4]. In animal studies excess of stable strontium demonstrated adverse effects on skeletal development in juveniles [5].In recent years, extraction chromatography appears to be simple, rapid and effective analytical technique for the separation of metal ion [6-7].

The discovery of crown ether opened a new era in supramolecular chemistry used in various fields in chemistry. As most of crown ether are useful as stationary phase in chromatographic method [8]. Polymeric crown ethers exbhits special features such as high resistance to chemicals, radiolysis, temperature and also polar solvents. By using poly [dibenzo-18-crown-6] we have reported the sorption behavior and selective separation of metal cation[9-11]. Horwitz et al. reported the chromatographic resin consisting of an octanol solution of 4,4'(5')-bis (t-butylcyclohexano)-18 -crown-6 sorbed on an inert polymeric support for Strontium (II)[9-12]. Macroporous silica-based N,N,N,N-tetraoctyl-3-oxapentane-1,5-diamide (TODGA) chelating polymeric used as chromatographic separation of Strontium (II)[13]. Systematic evaluations of a strontium-specific extraction chromatographic resin were carried out using crownether[14]. Reported method for separation alkaline earth metal[15]. Extraction behavior for strontium ions were carried out using various crown ether[16-23]. According to literature survey reveals that there are no reports on the use of poly [dibenzo-18-rown-6] for the chromatographic separation of Strontium (II) in glycine medium. The aim of the present study is to optimize conditions on a laboratory scale for the chromatographic separation and recovery of Strontium (II) from other associated metal ions employing poly [dibenzo-18-rown-6] as the sorbent in glycine as counter ion.

2.1 Experimental

Apparatus and reagents

A Ziess (German) Spectrophotometer, a digital pH meter (Model LI-120, ELICO, India) with glass and calomel electrodes and a digital Flame photometer (PI, Model No. 041, India) were used. A stock solution of strontium (II) was prepared by dissolving 1.207 g of strontium nitrate (Sigma-Aldrich Chemicals Pvt Limited Plot No 12 Bommasandra - Jigani Link Road 560100 BANGALORE INDIA) in 100 mL of distilled deionized water and standardized gravimetrically[24]. A solution containing 100 µg/mL of stronium(II) was prepared by appropriate dilution of standard stock solution. Picric acid solution (5×10^{-3} M) was prepared by dissolving 0.2846 g of picric acid in distilled demonized water and diluted to 250 mL.

Poly[dibenzo-18-crown-6] (E Merck Darmstadt, Germany) was used after screening to 100-200 mesh. A total of 0.5 g of polymer was slurred with distilled deionised water and poured into a Pyrex glass chromatographic column (20×0.8 cm i.d.). The column was used after preconditioning with Picric acid solution.

2.2 General procedure

100 µg of stronium(II) was mixed with picric acid in the concentration range of 1×10^{-4} M to 5×10^{-2} M in a total volume of 10 mL. The solution was then passed through poly [dibenzo-18-crown-6] column, preconditioning with same concentration of picric acid as that of the sample solution at flow rate of 0.5 mL/min. The column was then washed with the same concentration of picric acid. The sorbed strontium(II) was then eluted with different eluting agents (described later) at the flow rate of 0.5 mL/min. 5.0 mL fraction were collected and strontium(II) in the aqueous phase was determined spectrophotometrically with sulfonazo (III) at 640 nm[25]. The concentration of strontium(II) was calculated from a calibration graph.

3.1 Results and Discussion

Sorption of strontium(II) on poly[dibenzo-18-crown-6] as a function of picric acid concentration.

Sorption studies of strontium(II) were carried out from Picric acid medium. The concentration of picric acid was varied from 1×10^{-4} M to 5×10^{-2} M. After sorption, the elution of strontium(II) was carried out with 2.0 M hydrochloric acid. It was found that there was quantitative (100%) sorption of strontium (II) from 1×10^{-4} M to 5×10^{-2} M picric acid. The results are shown in (Figure: 1).

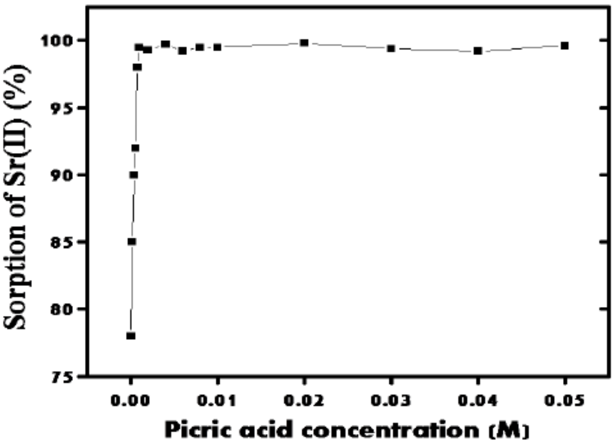

Figure 1. Sorption of strontium (II) as a function of picric acid concentration

The subsequent sorption studies of strontium(II) were carried out with 5×10^{-3} M picric acid .

3.2. Elution study of strontium(II) with various eluting agents

100 µg/mL of strontium(II) was sorbed on the poly[dibenzo-18-crown-6] column at 5×10^{-4} M picric acid concentration. After sorption, elution of strontium(II) was carried out using hydrochloric acid, hydrobromic acid, sulphuric acid, perchloric acid and acetic acid. The concentration of eluting agents varied from 0.5 M to 8.0 M. The elution profile of strontium(II) with various eluting agents is shown in (Figure : 2). Showed that strontium(II) was quantitatively eluted with 0.5 M to 6.0 M hydrochloric acid, 1.0 M sulphuric acid and 1.0 M hydrobromic acid whereas perchloric acid and acetic acid were found to be inefficient eluents for strontium(II). Further elution studies of strontium(II) in this work was carried out with 2.0 M hydrochloric acid.

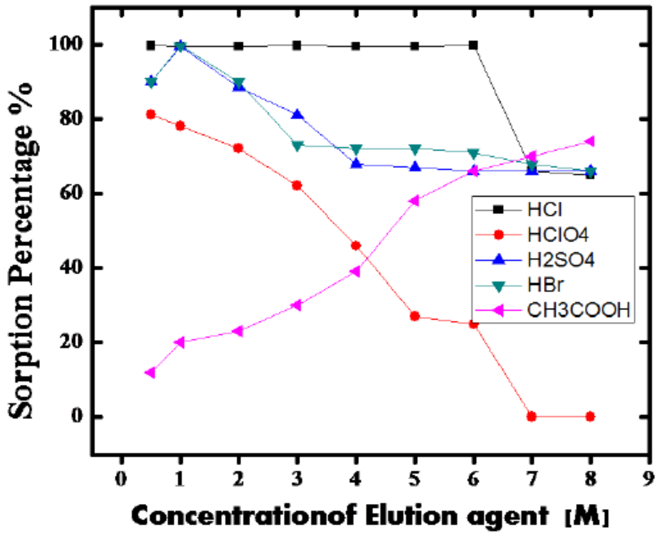

Figure: 2. Elution profile of strontium(II) with various eluting agents

3.3. Effect of varying concentration of strontium(II)

In order to find out the capacity of poly[dibenzo-18-crown-6] for the strontium(II), the concentration of strontium(II) was varied from 100-1000 µg/10 mL in picric acid and 2.0 M hydrochloric acid as eluent. The results (Table 3) showed that the sorption of strontium(II) was quantitative (99.6%) up to 600 µg. With increase in concentration of strontium(II) there was decrease in the percentage sorption of strontium(II) and is shown in the (Figure 3)From this study it was found that the capacity of poly[dibenzo-18-crown-6] for strontium(II) was found to be 1.74 ± 0.01 mmol/g of crown polymer (Figure: 3).

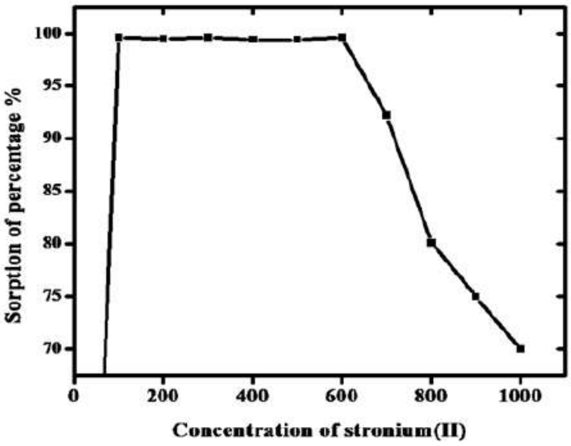

Figure 3 Effect of varying concentration of strontium(II)

3.4 Separation of strontium(II) from binary mixtures

An aliquot of solution containing 100 µg of strontium(II) was mixed with foreign ions and picric acid was added so that its concentration was 5×10^{-3} M in total volume of 10 mL. The tolerance limit was set as the amount of foreign ions required to cause ±2% deviation in the recovery of strontium(II). The solution was passed through a poly[dibenzo-18-crown-6] column, pre--conditioned with 5×10^{-3} M picric acid at a flow rate of 0.5 mL/min. Subsequently the column was washed with 15 mL of 5×10^{-3} M picric acid to remove unsorbed metal ions. Various foreign ions were not sorbed and hence passed through the column. The effluent was collected and analyzed for foreign ion content. The tolerance limit of various foreign ions is shown in Table 4.3.4. The most of the alkali metals show high tolerance limit except rubidium(I) and cesium(I). In case of alkaline metal lithium(I) cesium(I) and alkaline earth metals calcium(II), magnesium(II) tolerates strongly. Most of the p-block and d-block elements were sorbed and shows low tolerance limit. Amongst the inner transition elements, As compared lanthanum(III), cerium(III), thorium(III) low tolerance limit of uranium(VI) were sorbed quantitatively. The anion of inorganic and organic acids showed high tolerance limt (Table:1).

Table 1: Separation of strontium(II) from binary mixtures

Sr(II)- 100 µg, sorption- 1×10^{-4} M L-picric acid eluent- 2.0 M HCl

Ion	Added as	Tol. Limit (mg)	Ion	Added as	Tol. Limit (mg)
Li^+	LiCl	10	La^{3+}	$LaCl_3$	0.5
Na^+	NaCl	5.0	Ce^{3+}	$CeCl_3.6H_2O$	0.5
K^+	KCl	1.0	V^{5+}	NH_4SO_4	5.0
Rb^+	RbCl	5.0	Th^{4+}	$Th(NO_3)_4$	10
Cs^+	CsCl	15	U^{6+}	$UO_2(NO_3)_2.6H_2O$	0.3
Be^{2+}	$BeSO_4.4H_2O$	5.0	Mo^{6+}	$(NH_4)_6Mo_7O_{24}.4H_2O$	5.0
Mg^{2+}	$MgCl_2.6H_2O$	10	Cl^-	HCl	15
Ca^{2+}	$CaCl_2$	10	Br^-	HBr	15
Ba^{2+}	$Ba(NO_3)_2$	0.5	SCN^-	NaSCN	6.5
Co^{2+}	$CoCl_2.6H_2O$	0.5	ClO_4^-	$HClO_4$	15
Ni^{2+}	$NiCl_2.6H_2O$	0.5	CH_3COO^-	CH_3COOH	10
Mn^{2+}	$MnCl_2.4H_2O$	0.5	SO_4^{2-}	H_2SO_4	3.0
Zn^{2+}	$ZnCl_2$	5.0	BO_3^{3-}	H_3BO_3	15
Cr^{3+}	$Cr(NO_3)_3.9H_2O$	1.0	NO_3^-	HNO_3	5.0
Fe^{3+}	$FeCl_3.6H_2O$	1.0	Tartrate	Tartaric acid	10
Al^{3+}	$Al(NO_3)_3.9H_2O$	5.0	Ascorbate	Ascorbic acid	5.0

Table 2: Separation of strontium(II) from multicomponent mixture

No.	Mixture	Taken µg	Found* µg	Recovery %	Sorption Condition	Eluent
1	Li(I) Sr(II) Ba(II)	100 100 100	98.5 99.5 98.5	98.5 99.5 98.5	1×10^{-3} M Picric Acid	NSPC** 3.0 M HClO4. 0.5 M HCl
2	Mo(VI) Sr(II) Ba(II)	100 100 100	99.5 98.5 99.5	99.5 98.5 99.5	1×10^{-3} M Picric Acid	NSPC** 3.0 M HClO4. 0.5 M HCl
3	Fe(III) Sr(II) Ba(II)	100 100 100	98.5 99.5 98.5	98.5 99.5 98.5	1×10^{-3} M Picric Acid	NSPC** 3.0 M HClO4. 0.5 M HCl

* Average of triplicate analysis

** NSPC = No Sorption Passing through the Column

Ammonium carbonate

Table 3: Determination of strontium(II) in real sample

Sample	Present	Found
Milk	**30**	**29**
SY-II	**1100**	**1080**

4.3.3 Separation of strontium(II) from Multicomponent mixtures

Separation of strontium(II) was carried out from number of associated elements in multicomponent mixture. The mixture containing lithium(I), iron(III), barium(II), molybdenum(VI), strontium(II) was passed through the poly[dibenzo-18-crown-6] column at 1×10^{-4} M picric acid concentration, lithium(II), iron(III) was not sorbed and hence passed through the column. The barium(II), strontium(II), were sorbed. The sorbed strontium(II) was first eluted with 25 mL of 3.0 M perchoric acid. After that strontium(II) were eluted with 0.5 M acid and finally the barium(II) was eluted with 0.5 M hydrochloric acid and effluents are analyzed spectrophotometrically. Using this method, separation of barium(II), iron(III), lithium(II), strontium(II) mixtures was achieved. The results are shown in (Table: 2).

Application

(Table:3)

4.4 Conclusion

The important feature of this method is that using column chromatographic method and poly[dibenzo-18-crown-6] the separation of strontium(II) from associated element in picric acid medium has been achieved. The capacity of poly [dibenzo-18-crown-6] for strontium(II) was found to be 0.87 ± 0.01 mmol/g of crown polymer. Strontium(II) was separated from number of cations in binary as well as multicomponent mixtures. The method was extended to the determination of lead in real sample. The aim of present investigation is to demonstrate a simple, selective and cheap method and has good reproducibility (approximately ± 2%).

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Sorption Study of Nickel(II) in Glycine Medium Using Poly[dibenzo-18 crown-6] and Column Chromatography

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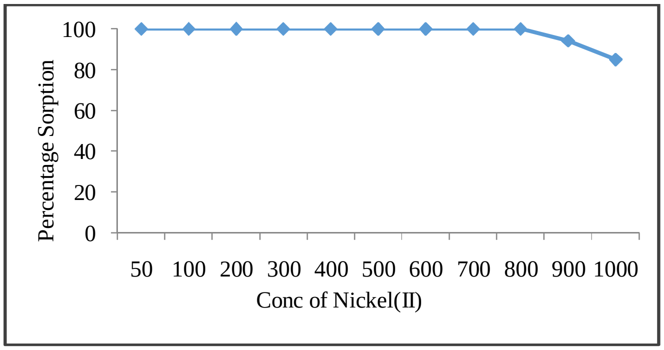
ABSTRACT

A simple chromatographic separation method has been developed for quantitative sorption of Ni(II) from an aqueous solution of 1×10^{-3} M Glycine using poly [dibenzo-18-crown-6] as stationary phase. The sorption of Ni(II) was quantitative 1×10^{-2}M to 1×10^{-6} M Glycine. The elution of Ni(II) was quantitative with 0.1–8.0 M HCl, 7.0-8.0M $HClO_4$, 5.0-8.0 M HBr M 0.1-8.0 M H_2SO_4 and 2.0-8.0M CH_3COOH The capacity of poly [dibenzo-18-crown-6] for Ni(II) was found to be 3.15 ± 0.01 mmol g^{-1} of crown polymer. The effects of concentrations of Glycine, Ni(II), foreign ions and eluents have been studied. Ni(II) was separated from a number of cations in multi component mixtures. The applicability of the proposed method was ascertained for the determination of Ni(II) in real samples. The reliability of method was checked by comparison of the results with those obtained using flame photometer. The method is very simple, rapid and selective with good reproducibility (approximately±2%).

Graphical Abstract



Effect of varying concentration of Ni(II)

Keywords: Sorption, Separation, Ni(II), Glycine, poly [dibenzo-18- crown-6], Chromatography.

INTRODUCTION

Nickel in nature is mainly available in the form of ores like Gernirite, Magnesium nickel silicate of variable composition. The important oxidation state of nickel is Ni(II). In olden days nickel is used as a coinage metal. Now days nickel becomes an important element, not only for industry, but also for biological systems. In rapidly expanding analytical fields, such as environmental, biological and material monitoring for trace metals. Nickel is used in ceramic industry and also used in batteries which are in convincing to the environment, dietary deficiency of nickel leading to various pathological manifestations. Nickel is relatively non toxic element. The high concentration of the nickel does show serious health hazards. The high incident of respiratory track moplacia and dermatitis has been observed when exposed by nickel refineries. In views of this separation and determination of nickel from associated elements is indispensable. Therefore, it is very important to develop sensitive, rapid and economical method for quantitative determination of it's trace amount in various samples of environmental importance. Several analytical techniques have been monitored for the determination of trace level Ni(II), it includes atomic absorption spectrometry[**1-4**] Inductive coupled plasma emission spectrometry[**5-7**] and X-Fluorence Spectrometry[**8, 9**] were reviewed. A few reagents are available for the spectrophotometric [**10-18**] determination of nickel (II).

Literature survey indicated that several spectrophotometric methods [**19-36**] were reported for the determination of Ni(II) by using various chromogenic reagents.

No attempts were made for the separation of nickel from associated element using amino acid media and column chromatography. This chapter describes in detail the sorption study and separation of Ni(II) using glycine medium on poly[dibenzo-18-crown-6. The concentration of glycine required for quantitative sorption of Ni(II) is very low, clean cut separation with good separation yield was achieved. The Ni(II) was successfully determined in various real samples by using this method.

MATERIALS AND METHODS

A Ziess (German) Spectrophotometer, a digital pH meter (Model LI-120, ELICO, India) with glass and calomel electrodes and a digital Flame photometer (PI, Model No. 041, India) were used. A stock solution of Ni(II) was prepared by dissolving 0.2808 g of nickel sulphate hepta hydrate (AR, Merck) in 100 mL of distilled deionized water and standardized gravimetrically using dimethyl glyoxime from complexometrically [**37**]. A solution containing 50 µg mL^{-1} of Ni(II) was prepared by appropriate dilution of standard stock solution. Glycine solution (1×10^{-1} M) was prepared by dissolving 1.875 g of glycine in distilled deionized water and diluted to 250 mL.

Poly[dibenzo-18-crown-6] (E Merck Darmstadt, Germany) was used after screening to 100-200 mesh. A total of 0.5 g of polymer was slurred with distilled deionised water and poured into a Pyrex glass chromatographic column (20×0.8 cm i.d.). The column was used after preconditioning with glycine solution.

50 µg of Nil(II) was mixed with glycine in the concentration range of 1×10^{-1} M to 1×10^{-9} M in a total volume of 10 mL. The solution was then passed through poly[dibenzo-18-crown-6] column, preconditioning with same concentration of glycine as that of the sample solution at flow rate of 0.5 mL min^{-1}. The column was then washed with the same concentration of glycine. The sorbed Nil(II) was then eluted with different eluting agents (described later) at the flow rate of 0.5 mL min^{-1}. 5.0 mL fraction were collected and Ni(II) in the aqueous phase was determined spectrophotometrically with dimethyl glyoxime at 445 nm. The concentration of Nil(II) was calculated from a calibration graph.

RESULTS AND DISCUSSION

Sorption of Ni(II) on poly[dibenzo-18-crown-6] as a function of glycine concentration: Sorption studies of Ni(II) were carried out from Glycine medium. The concentration of glycine was varied from 1×10^{-1} M to 1×10^{-9}M. After sorption, the elution of Ni(II) was carried out with 4.0 M hydrochloric acid. It was found that there was quantitative (100%) sorption of Ni(II) from 1×10^{-2} M to 1×10^{-6} M glycine. The results are shown in (Table 1) and (Figure 1). The subsequent sorption studies of Ni(II) were carried out with 1×10^{-3} M glycine.

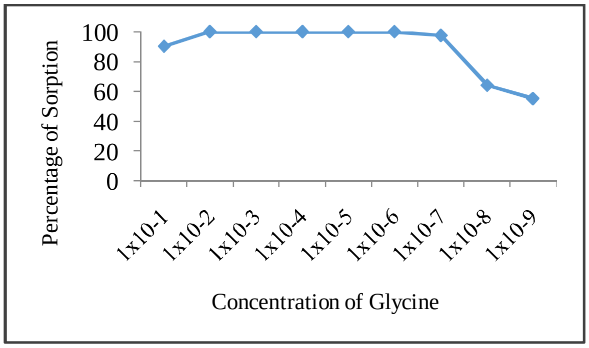


Figure 1. Sorption of Nil(II) as a function of glycine concentration Ni=50 µgmL^{-1}; Eluent= 4.0 M HCl.

Glycine concentration (M)	Sorption of Ni(II) (%)
1×10^{-1}	90.24
1×10^{-2}	100
1×10^{-3}	100
1×10^{-4}	100
1×10^{-5}	100
1×10^{-6}	100
1×10^{-7}	97.40
1×10^{-8}	64.20
1×10^{-9}	55.33

Table 1. Sorption of Nickel(II) as a function of glycine concentration Ni=50 µg mL^{-1}; Eluent= 4.0 M HCl

Elution study of Ni(II) with various eluting agents: 50 µg mL^{-1} of Nickel(II) was sorbed on the poly[dibenzo-18-crown-6] column at 1×10^{-3} M glycine concentration. After sorption, elution of Ni(II) was carried out using hydrochloric acid, hydrobromic acid, sulphuric acid, perchloric acid and acetic acid. The concentration of eluting agents varied from 0.1 M to 8.0 M. The elution profile of Ni(II) with various eluting agents is shown in (Figure 2). The results (Table 2) showed that Ni(II) was quantitatively eluted with 0.1 M to 8.0 M hydrochloric acid, 0.1 M to8.0 M sulphuric acid and 5.0 M to8.0 M hydrobromic acid, 7.0 M to 8.0 M Perchloric acid and 2.0 M to 8.0 M acetic acid. Further elution studies of Ni(II) in this work was carried out with 1.0 M hydrochloric acid.

Table 2. Elution of Nickel(II) with different eluting agents

Conc. Acid(M)	0.1M	0.5 M	1.0 M	2.0 M	3.0 M	4.0 M	5.0 M	6.0 M	7.0 M	8.0 M
	Percentage elution of Nickel(II)									
HCl	100	100	100	100	100	100	100	100	100	100
$HClO_4$	100	100	100	100	100	100	100	100	100	100
H_2SO_4	65.15	70.96	75.82	80.12	84.76	88.23	91.56	95.82	100	100
HBr	88.24	90.66	92.81	94.64	98.25	99.20	100	100	100	100
CH_3COOH	85.20	88.49	92.58	100	100	100	100	100	100	100

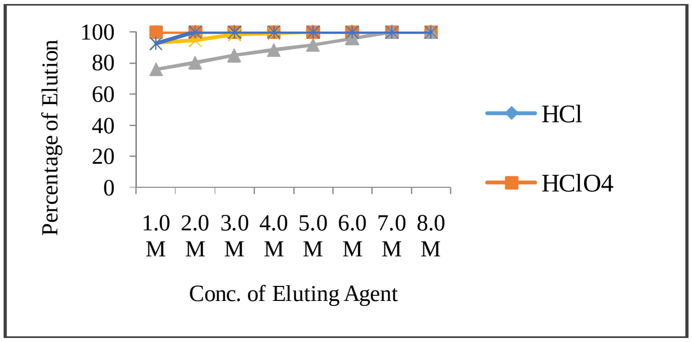


Figure 2. Elution profile of Ni(II) with various eluting agents

Effect of varying concentration of Ni(II): In order to find out the capacity of poly[dibenzo-18-crown-6] for the Ni(II), the concentration of Ni(II) was varied from 50-1000 µg $10mL^{-1}$ in glycine and 1.0 M hydrochloric acid as eluent. The results (Table 3) showed that the sorption of Ni(II) was quantitative (100%) up to 800 µg. With increase in concentration of Ni(II) there was decrease in the percentage sorption of Ni(II) and is shown in the (Figure 3) From this study it was found that the capacity of poly[dibenzo-18-crown-6] for Ni(II) was found to be 1.24 ± 0.01 mmol g^{-1} of crown polymer.

Concentration of Ni(II) (µg)	Percentage sorption
50	100
100	100
200	100
300	100
400	100
500	94.29
600	85.12
700	82.45
800	78.20

Table 3. Effect of varying concentration of Ni(II)

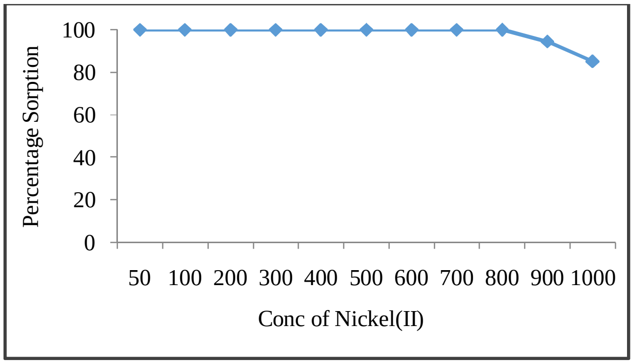


Figure 3. Effect of varying concentration of Ni(II)

Separation of Ni(II) from binary mixtures: An aliquot of solution containing 50 µg of Nickel(II) was mixed with foreign ions and glycine was added so that its concentration was 1×10^{-3} M in total volume of 10 mL. The tolerance limit was set as the amount of foreign ions required to cause ±2% deviation in the recovery of Ni(II). The solution was passed through a poly[dibenzo-18-crown-6] column, preconditioned with 1×10^{-3}M glycine at a flow rate of 0.5 mL min^{-1}. Subsequently the column was washed with 15 mL of 5×10^{-3}M glycine to remove unsorbed metal ions. Various foreign ions were not sorbed and hence passed through the column. The effluent was collected and analyzed for foreign ion content. The tolerance limit of various foreign ions is shown in table 4. The most of the alkali metals show high tolerance limit. In case of alkali metal lithium(I) cesium(I) and alkaline earth

Table 4. Separation of Ni(II) from binary mixtures Ni(II)- 50 µg,
sorption- 1×10^{-3} M L-glycinee eluent- 4.0 M HCl

Ion	Added as	Tol. Limit (mg)	Ion	Added as	Tol. Limit
Li^+	LiCl	24	Tl^{3+}	$Tl(NO_3)_3.3H_2O$	6
Na^+	NaCl	12	La^{3+}	$La(NO_3)_3.6H_2O$	4
K^+	KCl	8	Ce^{3+}	$CeCl_3.6H_2O$	1
Rb^+	RbCl	5	V^{4+}	$VOSO_4.4H_2O$	2
Cs^+	CsCl	30	Th^{4+}	$Th(NO_3)_4$	0.1
NH_4^+	NH_4Cl	16	Cr^{6+}	$K_2Cr_2O_7$	2.5
Be^{2+}	$BeSO_4.4H_2O$	5	Mo^{6+}	$(NH_4)_6Mo_7O_{24}.4H_2O$	0.3
Mg^{2+}	$MgCl_2.6H_2O$	18	W^{6+}	$Na_2WO_4O.4H_2O$	7
Ca^{2+}	$CaCl_2$	16	Cl^-	HCl	7
Sr^{2+}	$Sr(NO_3)_2$	3	Br-	HBr	11
Ba^{2+}	$Ba(NO_3)_2$	1	SCN^-	NaSCN	13
Co^{2+}	$CoCl_2.6H_2O$	0.6	ClO_4^-	$HClO_4$	7.4
Mn^{2+}	$MnCl_2.4H_2O$	0.5	CH_3COO^-	CH_3COOH	14
Zn^{2+}	$ZnCl_2$	5.8	SO_4^{2-}	H_2SO_4	9
U^{6+}	$UO_2(NO_3)_2.6H_2O$	10	BO_3^{3-}	H_3BO_3	4
Pb^{2+}	$Pb(NO_3)_2$	4.5	Tartrate	Tartaric acid	9
Cr^{3+}	$Cr(NO_3)_3.9H_2O$	0.1	EDTA	EDTA	2.5
Fe^{3+}	$FeCl_3.6H_2O$	1.7	Ascorbate	Ascorbic acid	8
Al^{3+}	$Al(NO_3)_3.9H_2O$	0.05			

metals calcium(II) and magnesium(II) tolerates strongly. Most of the p-block and d-block elements were sorbed and shows low tolerance limit. Amongst the inner transition elements chromium(VI) and aluminum(III) show low tolerance limit. The anion of inorganic and organic acids showed high tolerance limit (Table 4).

Separation of Ni(II) from Multicomponent mixtures: Separation of Ni(II) was carried out from number of associated elements in multi component mixture. The mixture containing lithium(I), zinc(II), lead(II), cadmium (II), thorium(IV), uranium(VI), magnesium (II), calcium (II) and strontium (II) was passed through the poly[dibenzo-18-crown-6] column at 1×10^{-3} M glycine concentration, lithium(I), magnesium (II), calcium (II) and strontium (II) was not sorbed and hence passed through the column. The zinc(II), lead(II), cadmium (II), thorium(IV), uranium(VI) were sorbed. The sorbed zinc(II) was first eluted with 25 mL of 0.5 M LiOH. After that Ni(II) were eluted with 4M hydrochloric acid and effluents are analyzed spectrophotometrically. Using this method separation of lead(II), cadmium (II), thorium(IV) and uranium(VI) mixtures was achieved. The results were shown in table 5.

Table 5. Separation of Ni(II) from multi component mixture

No.	Mixture	Taken µg	Found* µg	Recovery %	Sorption Condition	Eluent
1	Li(I)	50	49.5	90.0	1×10^{-3} M Glycine	NSPC**
	Zn(II)	40	39.9	99.80		0.5 M LiOH.
	Ni(II)	50	49.5	99.00		4.0 M HCl
2	Li(I)	50	50	100	1×10^{-3} M Glycine	NSPC**
	Pb(II)	40	39.8	99.50		0.2MA.C.
	Ni(II)	50	49.9	99.80		4.0 M HCl
3	Mg(II)	50	49.5	90.0	1×10^{-3} M Glycine	NSPC**
	Th(IV)	40	39.7	99.25		0.2M A.C.
	Ni(II)	50	49.8	99.60		4.0 M HCl
4	Li(I)	50	50	100	1×10^{-3} M Glycine	NSPC**
	Cd(II)	40	39.6	99.20		0.2MLiOH.
	Ni(II)	50	50	100		4.0 M HCl
5	Ca(II)	50	49.7	99.40	1×10^{-3} M Glycine	NSPC**
	Th(IV)	40	39.8	99.50		0.2M A.C.
	Ni(II)	50	49.8	99.60		4.0 M HCl
6	Sr(II)	50	49.6	99.20	1×10^{-3} M Glycine	NSPC**
	U(VI)	40	39.7	99.25		0.2M A.C.
	Ni(II)	50	49.8	99.60		4.0 M HCl
7	Mg(II)	50	49.7	99.40	1×10^{-3} M Glycine	NSPC**
	U(VI)	40	39.5	98.75		0.2M A.C.
	Ni(II)	50	49.9	99.80		4.0 M HCl

* Average of triplicate analysis

** NSPC = No Sorption Passing through the Column

APPLICATION

Determination of Ni(II) in Aluminium based alloy: About 0.4 g alloy samples was treated with 15 mL of 1:1 HCl, to this 3 mL of HNO_3 was added and the contents boiled until dissolution was complete. Then, 10 mL of water and 40 mL of 4N ammonium hydroxide solution were added and filtered through a Whatman filter paper (No.41). The filtrate was collected into 25 mL volumetric flask and made up to the marks with distilled water.

Sample	Present%	Found %
BAS-20	1.93	1.91
BAS-85	0.91	0.90
Cu-Ni alloy	31.20	31.15

Determination of Ni(II) in Vegetable Oils: 100g of hydrogenated groundnut oil (edible) was dried in a hot air oven at 100°C and subsequently dissolved in 20ml mixture of 1:2:5 H_2SO_4:H_3PO_4:HNO_3. The contents were heated until sulphurous fumes were evolved and the volume was reduced to about 5 mL. A little quantity of distilled water was added and filtered through an acid washed Whatman 41 filter paper into a 100 mL volumetric flask and made up to the mark with distilled water.

Sample	Present µg mL^{-1}	Found µg mL^{-1}
Hydrogenated Groundnut Oil	0.56	0.55

CONCLUSION

The important feature of this method is that using column chromatographic method and poly[dibenzo-18-crown-6] the separation of Ni(II) from associated element in Glycine medium has been achieved. The capacity of poly[dibenzo-18-crown-6] for Nickel(II) was found to be 1.24 ± 0.01 mmol g^{-1} of crown polymer. Ni(II) was separated from number of cations in binary as well as multi component mixtures. The method was extended to the determination of nickel in real sample. The aim of present investigation is to demonstrate a simple, selective and cheap method and has good reproducibility (approximately ± 2%).

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Sorption Study of Chromium(III) in Glycine Medium Using Poly[dibenzo-18 crown-6] and Column Chromatography

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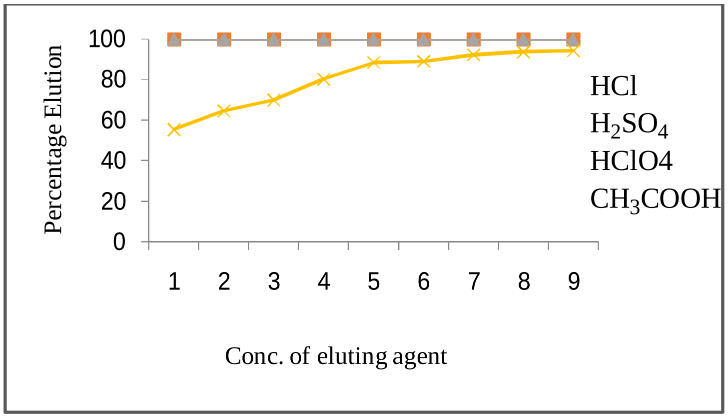
ABSTRACT

A simple chromatographic separation method has been developed for quantitative sorption of chromium(III) from an aqueous solution of 1×10^{-5} *M Glycine using poly [dibenzo-18-crown-6] as stationary phase. The sorption of chromium(III) was quantitative* 1×10^{-3}*M to* 1×10^{-6}*M Glycine. The elution of chromium(III) was quantitative with 0.5–8.0 M HCl, 0.5-8M* $HClO_4$ *and 0.5-8.0 M* H_2SO_4*. The capacity of poly [dibenzo-18-crown-6] for chromium(III) was found to be 1.54 ± 0.01 mmol/g of crown polymer. The effects of concentrations of glycine, chromium(III), foreign ions and eluents have been studied. Chromium(III) was separated from a number of cations in Multi component mixtures. The applicability of the proposed method was ascertained for the determination of chromium(III) in real samples. The reliability of method was checked by comparison of the results with those obtained using flame photometer. The method is very simple, rapid and selective with good reproducibility (approximately±2%).*

Graphical Abstract



Elution of chromium(III) with various eluting agents

Keywords: Sorption, Separation, Chromium(III), Chromatography, poly [dibenzo-18-crown-6], Glycine.

INTRODUCTION

Chromium and its compounds are very useful in everyday life. It is used on a large scale in many different industries, including metallurgical, electroplating, production of paints and pigments, tanning, wood preservation, chromium chemicals production, and pulp and paper production. The recovery and removal of such species are important not only from environment protection view point, but also for the importance as the secondary resource recycling and economical purposes [1]. Chromium and its compounds are known as strategic minerals widely used in various industrial plants such as electroplating, leather tanning, painting, pigment production and metallurgy [1, 2]. Chromium metal (Cr) occurs naturally in the environment and has both beneficial and potential human risks. Cr exists in many oxidation states with Cr(III) and Cr(VI) being the primary existing oxidation states in the environment. Cr(III)is an essential nutrient for maintaining lipid, insulin, and glucose metabolism and its deficiency may lead to diabetes [3], of the many Cr species, hexavalent chromium (Cr(VI)) is one of the most toxic, especially when compared to trivalent chromium [4]. Chromium oxidation states ranges from +2 to +6. Among them trivalent and hexavalent ions are two relatively stable valance states in the natural samples [5]. Although Cr(III) is an essential component for some biological activities, Cr(VI) is categorized as one of the most toxic chemical species, being an agent for various cancers [2, 6]. Thus, there moving of hexavalent chromium from industrial wastewater before discharging to the environment is a crucial concern as well as a challenging problem in industries from economical view point. Liquid-liquid extraction [7-10], facilitated transport through liquid membrane [11-14], adsorption by activated carbon [15-18] and ion exchange [19-22] are among the techniques frequently used for recovering of chromium ions. Nowadays, membrane technologies have gained an important role in a variety of industries such as water desalination, food processing and medical applications [14, 23, 24].

Many methods have been reported for the quantitative determination of chromium. The analytical technique varies from inductively coupled plasma-atomic emission spectroscopy [25], atomic absorption spectroscopy [26], neutron activation analysis [27], X-ray absorption spectroscopy [28], complexometric [29], catalytic kinetic [30], sequential injection [31] to flow injection methods [32-35], for the separation of chromium various methods were used [36-38].

According to literature survey reveals that there are no reports on the use of poly[dibenzo-18-crown-6] for the chromatographic separation of chromium(III)in glycine medium. The aim of the present study is to optimize conditions on a laboratory scale for the chromatographic separation and recovery of chromium(III) from other associated metal ions employing poly[dibenzo-18-crown-6] as the sorbent in glycine as counter ion.

MATERIALS AND METHODS

Apparatus and reagents: A Ziess (German) Spectrophotometer, a digital pH meter (Model LI-120, ELICO, India) with glass and calomel electrodes and a digital Flame photometer (PI, Model No. 041, India) were used.

All chemicals were of analytical reagent grade or chemically pure grade and distilled water was used throughout the study. Chromium sulphate [$Cr_2(SO_4)_3$](E Merck grade) was used for preparing chromium(III) stock solution (1M) and standardized gravimetrically [39]. A solution containing 100 µg mL^{-1} of chromium(III) was prepared by appropriate dilution of standard stock solution. Glycine solution (1×10^{-1} M) was prepared by dissolving 1.875 g of glycine in distilled demonized water and diluted to 250 mL. Poly[dibenzo-18-crown-6] (E Merck Darmstadt, Germany) was used after screening to 100-200 mesh. A total of 0.5 g of polymer was slurred with distilled deionized water and poured into a Pyrex glass chromatographic column (20×0.8 cm i.d.). The column was used after preconditioning with glycine solution.

General procedure: 50 μg of chromium(III) was mixed with glycine in the concentration range of 1×10^{-1} M to 1×10^{-9} M in a total volume of 10 mL. The solution was then passed through poly [dibenzo-18-crown-6] column, preconditioning with same concentration of glycine as that of the sample solution at flow rate of 0.5 mL min^{-1}. The column was then washed with the same concentration of glycine. The sorbed chromium(III) was then eluted with different eluting agents (described later) at the flow rate of 0.5 mL min^{-1}. 5.0 mL fraction were collected and chromium(III) in the aqueous phase was determined spectrophotometrically. Takao *et al*., described a extraction-spectrophotometric determination of with 4-(2-pyridylazo)-resorcinol (PAR) at 540 nm [**40**]. The concentration of chromium(III) was calculated from a calibration graph.

RESULTS AND DISCUSSION

Sorption of chromium(III)on poly[dibenzo-18-crown-6] as a function of glycine concentration: Sorption studies of chromium(III) were carried out from glycine medium. The concentration of glycine was varied from 1×10^{-1} M to 1×10^{-9} M (Table 1). After sorption, the elution of chromium(III) was carried out with 4.0 M hydrochloric acid. It was found that there was quantitative (100%) sorption of chromium(VI) from 1×10^{-3} M to 1×10^{-6} M glycine. The results are shown in **(**Figure 1**).** The subsequent sorption studies of chromium(III) were carried out with 1×10^{-3} M glycine.

Table 1. Sorption of chromium(III) as a function of glycine concentration
Cr(III) = 50 μg mL^{-1}, Eluent = 4.0 M HCl

Glycine concentration (M)	Sorption of Cr(VI) (%)
$1\text{x}10^{-1}$	88.20
$1\text{x}10^{-2}$	97.34
$1\text{x}10^{-3}$	100
$1\text{x}10^{-4}$	100
$1\text{x}10^{-5}$	100
$1\text{x}10^{-6}$	100
$1\text{x}10^{-7}$	94.25
$1\text{x}10^{-8}$	88.55
$1\text{x}10^{-9}$	76.20

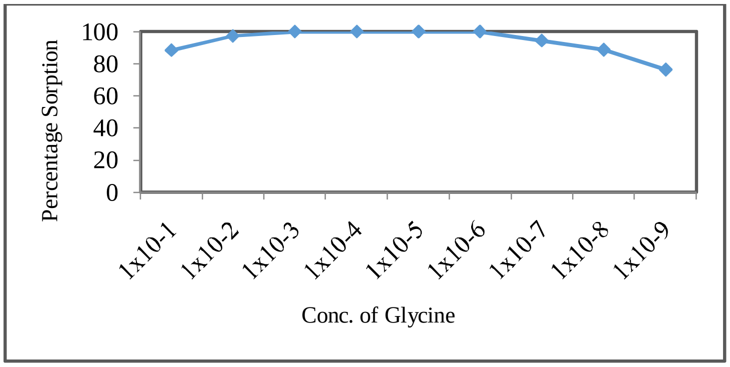


Figure 1. Sorption of chromium(III) as a function of glycine concentration

Elution study of chromium(III) with various eluting agents: 50 μg mL^{-1} of chromium(III) was sorbed on the poly[dibenzo-18-crown-6] column at 1×10^{-5}M glycine concentration. After sorption, elution of chromium(III) was carried out using hydrochloric acid, sulphuric acid, perchloric acid and acetic acid (Table 2). The concentration of eluting agents varied from 0.5 M to 8.0 M. The elution profile of chromium(III) with various eluting agents is shown in (Figure 2). Showed that chromium(III) was quantitatively eluted with 0.5 M to 8.0 M hydrochloric acid and Sulphuric acid, 0.5 M to 8.0 M perchloric acid. Further elution studies of chromium(III) in this work was carried out with 4.0 M hydrochloric acid.

Table 2.Elution of chromium(III) with different eluting agents Cr(III) = 50 µg mL^{-1}, glycine $1\times10^{-5}M$

Conc. Acid	0.5	1.0	2.0	3.0	4.0	5.0	6.0	7.0	8.0
	Percentage Elution								
HCl	100	100	100	100	100	100	100	100	100
H_2SO_4	100	100	100	100	100	100	100	100	100
$HClO_4$	100	100	100	100	100	100	100	100	100
CH_3COOH	55.60	64.82	70.12	80.45	88.60	89.18	92.45	93.86	94.45

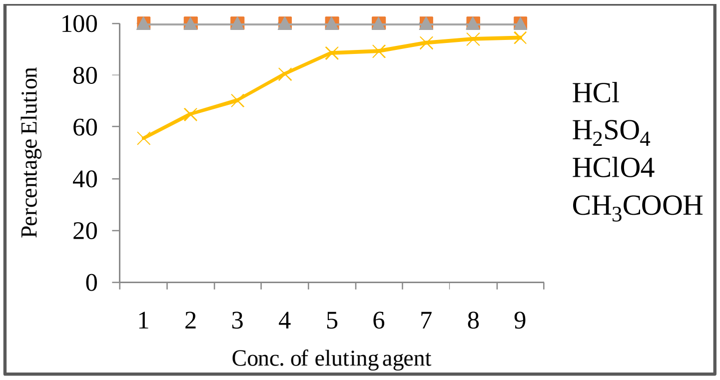


Figure 2. Elution of chromium(III) with various eluting agents.

Effect of varying concentration of chromium(III): In order to find out the capacity of poly [dibenzo-18-crown-6] for the chromium(III), the concentration of chromium(III) was varied from 50-600 µg $10mL^{-1}$ in glycine and 4.0 M hydrochloric acid as eluent. The results (Table 3) showed that the sorption of chromium(III) was quantitative (100%) up to 400 µg. With increase in concentration of chromium(III) there was decrease in the percentage sorption of chromium(III) and is shown in the (Figure 3). From this study it was found that the capacity of poly[dibenzo-18-crown-6] for chromium(III) was found to be 1.54 ± 0.01 mmol g^{-1} of crown polymer.

Table 3. Effect of varying concentration of chromium (III) glycine $1x10^{-5}$ M, eluent 1.0 M HCl

Chromium(III) (µg)	Percentage sorption (%)
50	100
100	100
150	100
200	100
250	100
300	100
350	100
400	100
450	97.30
500	95.45
550	84.46
600	77.51

Separation of chromium(III) from binary mixtures: An aliquot of solution containing 50 µg of chromium(III) was mixed with foreign ions and glycine was added so that its concentration was 1×10^{-5} M in total volume of 10 mL. The tolerance limit was set as the amount of foreign ions required to cause ±2% deviation in the recovery of chromium(III). The solution was passed through a poly[dibenzo-18-crown-6] column, preconditioned with 1×10^{-5} M glycine at a flow rate of 0.5 mL min^{-1}. Subsequently the column was washed with 15 mL of 1×10^{-5} M glycine to remove unsorbed metal ions. Various foreign ions were not sorbed and hence passed through the column. The effluent was collected and analyzed for foreign ion content. The tolerance limit of various foreign ions is shown in table 4. The most of the alkali metals show high tolerance limit. In case of alkaline metal lithium(I), sodium(I), potassium (I), rubidium(I) and cesium(I) are strongly tolerates. Most of the p-block and d-block elements were sorbed and shows low tolerance limit. Amongst the inner transition elements, as compared lanthanum(III), cerium(III), thallium(III) show low tolerance limit. The anion of inorganic and organic acids showed high tolerance limit (Table 4).

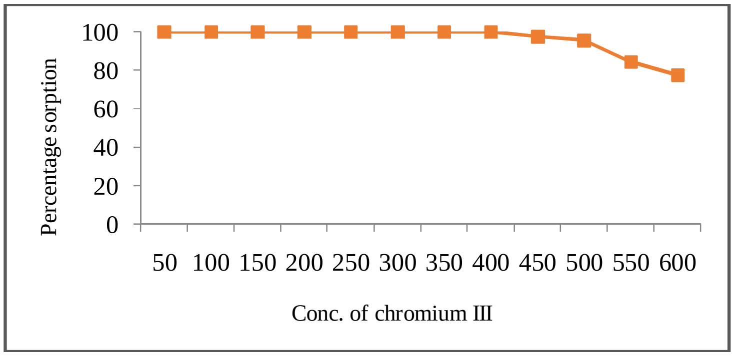


Figure 3. Effect of varying concentration of chromium(III).

Table 4. Separation of chromium(III) from binary mixtures Cr(III)- 50 µg, sorption- 1×10^{-5} M glycine, eluent- 2.0 M HCl

Ion	Added as	Tol. Limit (mg)	Ion	Added as	Tol. Limit
Li^+	LiCl	15	Tl^{3+}	$Tl(NO_3)_3.3H_2O$	0.1
Na^+	NaCl	12	La^{3+}	$La(NO_3)_3.6H_2O$	0.03
K^+	KCl	18	Ce^{3+}	$CeCl_3.6H_2O$	0.8
Rb^+	RbCl	16	V^{4+}	$VOSO_4.4H_2O$	0.7
Cs^+	CsCl	8	Th^{4+}	$Th(NO_3)_4$	0.8
NH_4^+	NH_4Cl	14	Cr^{6+}	$K_2Cr_2O_7$	1.3
Be^{2+}	$BeSO_4.4H_2O$	1.5	Mo^{6+}	$(NH_4)_6Mo_7O_{24}.4H_2O$	9
Mg^{2+}	$MgCl_2.6H_2O$	0.5	W^{6+}	$Na_2WO_4O.4H_2O$	7
Ca^{2+}	$CaCl_2$	1.5	Cl^-	HCl	2
Sr^{2+}	$Sr(NO_3)_2$	0.1	Br-	HBr	7
Ba^{2+}	$Ba(NO_3)_2$	0.8	SCN^-	NaSCN	3
Co^{2+}	$CoCl_2.6H_2O$	Co-extran	ClO_4^-	$HClO_4$	3
Ni^{2+}	$NiCl_2.6H_2O$	Co-extran	CH_3COO^-	CH_3COOH	4
Zn^{2+}	$ZnCl_2$	0.5	$SO4\ _2^-$	H_2SO_4	2
U^{6+}	$UO_2(NO_3)_2.6H_2O$	2	$BO_3\ ^{3-}$	H_3BO_3	6
Pb^{2+}	$Pb(NO_3)_2$	4	Tartrate	Tartaric acid	11
Fe^{3+}	$FeCl_3.6H_2O$	1.2	EDTA	EDTA	6
Al^{3+}	$Al(NO_3)_3.9H_2O$	0.5	Ascorbate	Ascorbic acid	3

Separation of chromium(III) from Multicomponent mixtures: Separation of chromium(III) was carried out from number of associated elements in multi component mixture. The mixture containing lithium(I), uranium(VI), chromium(III), cobalt(II), mercury(II), potassium(I), sodium(I), nickel (II), manganese(II), cadmium(II) and lead(II) was passed through the poly[dibenzo-18-crown-6] column at 1×10^{-3} M glycine concentration, lithium(I), potassium (I), sodium(I) was not sorbed and hence passed through the column. The uranium(VI), chromium(III), cobalt(II) mercury(II), potassium(I), nickel (II), manganese(II), cadmium(II) and lead(II) were sorbed. The sorbed uranium(VI) was first eluted with 25 mL of 0.2 M ammonium carbonate. After that chromium(III) were eluted with 4 M hydrochloric acid and effluents are analyzed spectrophotometrically. Using this method, separation of cobalt(II), chromium(III), mercury(II), potassium(I), manganese(II), nickel (II), lithium(I), potassium (I), sodium(I), cadmium(II) and lead(II) mixtures was achieved. The results are shown in table 5.

Table 5. Separation of chromium(III) from Multicomponent mixtures

No	Mixture	Taken µg	Found µg	Recovery* %	Sorption Condition	Eluent
1	Li(I)	100	100	100	1x10^{-3}M Glycine	NSPC**
	U(VI)	100	100	100		0.2 A.C.
	Cr(III)	50	49	98		4 M HCl
2	K(I)	100	99	99	1x10^{-3} M Glycine	NSPC**
	Cr(III)	50	50	100		4 M HCl
	Co(II)	50	48	96		0.5 M HBr
3	Na(I)	100	100	100	1x10^{-3}M Glycine	NSPC**
	Cr(III)	50	49.5	99		4 M HCl
	Hg(II)	100	98	98		0.5 M HBr
4	Li(I)	100	100	100	1x10^{-3}M Glycine	NSPC**
	Pb(II)	25	24	96		3 M HBr
	Cr(III)	50	49	98		3.0 M HCl
5	Li(I)	100	100	100	1x10^{-3} M Glycine	NSPC**
	Mn (II)	50	49	98		0.5 M HBr
	Cr(III)	50	50	100		3.0M HCl
6	K(I)	100	99	99	1x10^{-3} M Glycine	NSPC**
	Cd(II)	50	49	98		0.5 M HBr
	Cr(III)	50	49.5	99		3.0M HCl
7	Li(I)	100	100	100	1x10^{-3} M Glycine	NSPC**
	Ni(II)	50	49	98		0.5 M HBr
	Cr(III)	50	49.5	99		3.0M HCl

* Average of triplicate analysis, ** NSPC = No Sorption Passing through the Column
A.C.- Ammonium Carbonate

APPLICATION

Determination of chromium in pharmaceuticals samples: The sample of tablet was dissolved in 10 mL 0.1 M HCl. The solution was evaporated almost to dryness it was lixiviated with water and solution was made up to 500 mL aliquot of solution was taken from which chromium(III)was extracted and analyzed as per general procedure (Table 6).

Table 6. Determination of chromium in pharmaceuticals samples

Sample	Present Mcg tablet^{-1}	Found Mcg tablet^{-1}
Chromoplex	0.200	0.199
Fourts B	0.150	0.149
Optisulin	0.500	0.498

CONCLUSION

The important feature of this method is that using column chromatographic method and poly[dibenzo-18-crown-6] the separation of chromium(III) from associated element in glycine medium has been achieved. The capacity of poly [dibenzo-18-crown-6] for chromium(III) was found to be 1.54 ± 0.01 mmol/g of crown polymer. Chromium(III) was separated from number of cations in binary as well as multi component mixtures. The method was extended to the determination of chromium(III) in real sample. The aim of present investigation is to demonstrate a simple, selective and cheap method and has good reproducibility (approximately ± 2%).

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Sorption study of Cadmium(II) in glycine medium using poly [dibenzo-18 crown-6] and Column chromatography

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Abstract

A simple chromatographic separation method has been developed for quantitative sorption of Cadmium(II) from an aqueous solution of 1×10^{-3} M Glycine using poly [dibenzo-18-crown-6] as stationary phase. The sorption of Cadmium(II) was quantitative 1×10^{-2}M to 1×10^{-4}M Glycine. The elution of Cadmium(II) was quantitative with 1.0–8.0 M HCl, 1.0-8.0M $HClO_4$, 2.0-8.0 M H2SO4 and 1.0-8.0M CH3COOH The capacity of poly [dibenzo-18-crown-6] for Cadmium(II) was found to be 0.533 ± 0.01 mmol/g of crown polymer. The effects of concentrations of Glycine, Cadmium (II), foreign ions and eluents have been studied. Cadmium(II) was separated from a number of cations in multicomponent mixtures. The applicability of the proposed method was ascertained for the determination of Cadmium(II) in real samples. The reliability of method was checked by comparison of the results with those obtained using flame photometer. The method is very simple, rapid and selective with good reproducibility (approximately±2%).

Keywords: sorption, separation, Cadmium(II), Glycine, poly [dibenzo-18- crown-6], Chromatography

1. Introduction

Cadmium makes up about 0.1 ppm of Earth's crust. Compared with the more abundant 65 ppm zinc, cadmium is rare [1]. No significant deposits of cadmium-containing ores are known Greenockite (CdS), the only cadmium mineral of importance, is nearly always associated with sphalerite (ZnS). This association is caused by the geochemical similarity between zinc and cadmium which makes geological separation unlikely. As a consequence, cadmium is produced mainly as a byproduct from mining, smelting, and refining sulfidic ores of zinc, and, to a lesser degree, lead and copper. Small amounts of cadmium, about 10% of consumption, are produced from secondary sources, mainly from dust generated by recycling iron and steel scrap. Production in the United States began in 1907 [2] but it was not until after World War I that cadmium came into wide use [3, 4]. One place where metallic cadmium can be found is the Vilyuy River basin in Siberia [5].

A considerable amount of work has been done in recent years on the ion exchange separation of cadmium from various other elements [6, 17]. While Kallmann's [11] anion exchange method has successfully separated a considerable number of cations from cadmium, published methods on cation exchange have been applied to separate cadmium from only a small number of other cations, and are therefore of a limited application.

The discovery of crown ether opened a new era in supramolecular chemistry used in various fields in chemistry. As most of crown ether are useful as stationary phase in chromatographic method [18]. Polymeric crown ethers exhibit special features such as high resistance to chemicals, radiolysis, temperature and also polar solvents. By using poly [dibenzo-18-crown-6] we have reported the sorption behavior and selective separation of metal cation [19, 23]. According to literature survey reveals that there are no reports on the use of poly [dibenzo-18-rown-6] for the chromatographic separation of Cadmium(II) in glycine medium. The aim of the present study is to optimize conditions on a laboratory scale for the chromatographic separation and recovery of Cadmium(II) from other associated metal ions employing poly [dibenzo-18-crown-6] as the sorbent in glycine as counter ion.

2 Experimental

2.1 Apparatus and reagents

A Ziess (German) Spectrophotometer, a digital pH meter (Model LI-120, ELICO, India) with glass and calomel electrodes and a digital Flame photometer (PI, Model No. 041, India) were used. A stock solution of Cadmium(II) was prepared by dissolving 1.207 g of cadmium nitrate (Sigma-Aldrich Chemicals Pvt Limited Plot No 12 Bommasandra - Jigani Link Road 560100 Bangalore India) in 100 mL of distilled deionized water and standardized gravimetrically [24]. A solution containing 100 µg/mL of Cadmium(II) was prepared by appropriate dilution of standard stock solution. Glycine solution (1×10^{-1} M) was prepared by dissolving 1.875 g of glycine in distilled deionized water and diluted to 250 mL. Poly [dibenzo-18-crown-6] (E Merck Darmstadt, Germany) was used after screening to 100-200 mesh. A total of 0.5 g of polymer was slurred with distilled deionised water and poured into a Pyrex glass chromatographic column (20×0.8 cm i.d.). The column was used after preconditioning with glycine solution.

2.2 General procedure

50 µg of Cadmium(II) was mixed with glycine in the concentration range of 1×10^{-7} M to 1×10^{-1}M in a total volume of 10 mL. The solution was then passed through poly [dibenzo-18-crown-6] column, preconditioning with same

concentration of glycine as that of the sample solution at flow rate of 0.5 mL/min. The column was then washed with the same concentration of glycine. The sorbed Cadmium(II) was then eluted with different eluting agents (described later) at the flow rate of 0.5 mL/min. 5.0 mL fraction were collected and Cadmium(II) in the aqueous phase was determined spectrophotometric ally with PAR at 520 nm [25]. The concentration of Cadmium(II) was calculated from a calibration graph.

3. Results and Discussion

3.1. Sorption of Cadmium(II) on poly [dibenzo-18-crown-6] as a function of glycine concentration.

Sorption studies of Cadmium(II) were carried out from glycine medium. The concentration of glycine was varied from 1×10^{-7} M to 1×10^{-1}M. After sorption, the elution of Cadmium(II) was carried out with 4.0 M hydrochloric acid. It was found that there was quantitative (100%) sorption of Cadmium(II) from 1×10^{-4} M to 1×10^{-2} M L- glycine. The results are shown in (Figure: 1). the subsequent sorption studies of Cadmium(II) were carried out with 5×10^{-3} M glycine.

Table 1: Sorption of Cadmium(II) as a function of glycine concentration Cd (II) = 50 µg, Eluent = 4.0 M HCl

Glycine concentration (M)	Sorption of Cd (II) (%)
$1x10^{-1}$	85.4
$1x10^{-2}$	100
$1x10^{-3}$	100
$1x10^{-4}$	100
$1x10^{-5}$	88
$1x10^{-6}$	86
$1x10^{-7}$	65

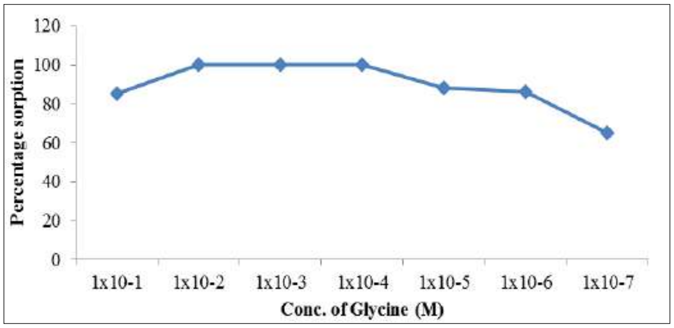

Fig 1: Sorption of Cadmium(II) as a function of glycine concentration.

3.2. Elution study of Cadmium(II) with various eluting agents

50 µg/mL of Cadmium(II) was sorbed on the poly [dibenzo-18-crown-6] column at 5×10^{-4} M glycine concentration. After sorption, elution of Cadmium(II) was carried out using hydrochloric acid, sulphuric acid, perchloric acid and acetic acid. The concentration of eluting agents varied from 0.5 M to 8.0 M. The elution profile of Cadmium(II) with various eluting agents is shown in (Fig 2). It showed that Cadmium(II) was quantitatively eluted with 1.0 M to 8.0 M hydrochloric acid, 2.0M to 8.0 M sulphuric acid, 1.0M to 8.0 M per chloric acid and 1.0M to 8.0 M acetic acid. Further elution studies of Cadmium(II) in this work was carried out with 4.0 M hydrochloric acid.

Table 2: Elution of Cadmium(II) with different eluting agents Cd (II) = 50 µg, glycine 0.001 M

Conc.	0.5	1.0	2.0	3.0	4.0	5.0	6.0	7.0	8.0
Acid	Percentage Elution (%)								
HCl	96	100	100	100	100	100	100	100	100
H_2SO_4	90	94	100	100	100	100	100	100	100
$HClO_4$	96	100	100	100	100	100	100	100	100
CH_3COOH	98	100	100	100	100	100	100	100	100

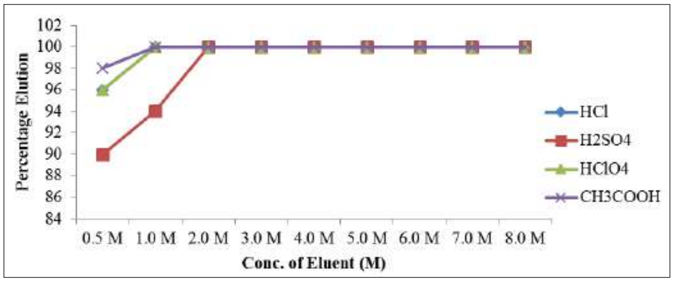

Fig 2: Elution of Cadmium(II) with different eluting agents.

3.3. Effect of varying concentration of Cadmium (II)

In order to find out the capacity of poly [dibenzo-18-crown-6] for the cadmium (II), the concentration of Cadmium(II) was varied from 100-1000 µg/10 mL in glycine and 4.0 M hydrochloric acid as eluent. The results (Table 3.3) showed that the sorption of Cadmium(II) was quantitative (100%) up to 300 µg. With increase in concentration of Cadmium(II) there was decrease in the percentage sorption of Cadmium(II) and is shown in the Fig 3. From this study it was found that the capacity of poly [dibenzo-18-crown-6] for Cadmium(II) was found to be 0.944 ± 0.01 mmol/g of crown polymer.

Table 3: Effect of varying concentration of Cadmium(II) glycine 1×10^{-3} M, eluent 1.0 M HCl

Cadmium(II) (µg)	Percentage sorption (%)
25	100
50	100
75	100
100	100
125	100
150	100
175	100
200	100
225	100
250	100
275	100
300	100
325	93
350	86
375	72
400	68
450	63
475	60

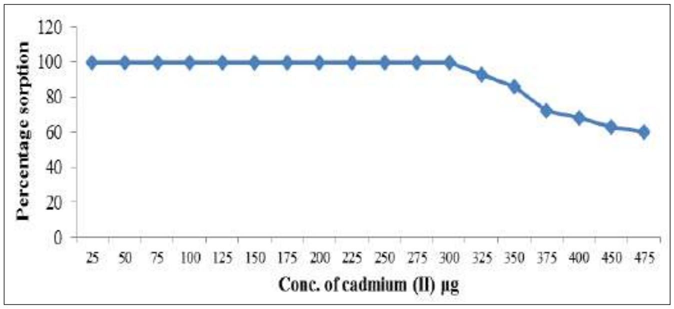

Fig 3: Effect of varying concentration of cadmium (II).

3.4 Separation of Cadmium(II) from binary mixtures

An aliquot of solution containing 50 µg of Cadmium(II) was mixed with foreign ions and glycine was added so that its concentration was 1×10^{-3} M in total volume of 10 mL. The tolerance limit was set as the amount of foreign ions required to cause ±2% deviation in the recovery of cadmium (II). The solution was passed through a poly [dibenzo-18-crown-6] column, preconditioned with 1×10^{-3}M glycine at a flow rate of 0.5 mL/min. Subsequently the column was washed with 15 mL of 1×10^{-3}M glycine to remove un sorbed metal ions. Various foreign ions were not sorbed and hence passed through the column. The effluent was collected and analyzed for foreign ion content. The tolerance limit of various foreign ions is shown in Table 4. The most of the alkali metals show high tolerance limit. In case of alkaline metal lithium(I), sodium(I), potassium(I), rubidium(I), cesium(I) and alkaline earth metals magnesium(II), calcium(II), strontium(II) tolerate strongly. Most of the p-block and d-block elements were sorbed and shows low tolerance limit. Amongst the inner transition elements, as compared lanthanum (III), thallium (IV), cerium (III), shows low tolerance limit. The anion of inorganic and organic acids showed high tolerance limit.

Table 4: Separation of Cadmium(II) from binary mixtures Cd(II) = 50 µg, sorption- 1×10^{-3} M glycine, eluent- 4.0 M HCl

Ion	Added as	Tol. Limit (mg)	Ion	Added as	Tol. Limit
Li^+	LiCl	10	Tl3+	$Tl(NO_3)_3.3H_2O$	0.8
Na^+	NaCl	9	La3+	$La(NO_3)_3.6H_2O$	0.5
K^+	KCl	7	Ce^{3+}	$CeCl_3.6H_2O$	0.5
Rb^+	RbCl	6	V^{4+}	$VOSO_4.4H_2O$	0.5
Cs^+	CsCl	7	Th^{4+}	$Th(NO_3)_4$	Co-Extrn
NH_4^+	NH_4Cl	15	Cr^{6+}	$K_2Cr_2O_7$	0.4
Be^{2+}	$BeSO_4.4H_2O$	Co-Extrn	Mo^{6+}	$(NH_4)_6Mo_7O_{24}.4H_2O$	2.5
Mg^{2+}	$MgCl_2.6H_2O$	5	W6+	$Na_2WO_4O.4H_2O$	6
Ca^{2+}	$CaCl_2$	5	Cl^-	HCl	5
Sr^{2+}	$Sr(NO_3)_2$	2	Br-	HBr	3
Ba^{2+}	$Ba(NO_3)_2$	0.8	SCN^-	NaSCN	4
Co^{2+}	$CoCl_2.6H_2O$	Co-Extrn	ClO_4^-	$HClO_4$	1
Ni^{2+}	$NiCl_2.6H_2O$	Co-Extrn	CH_3COO^-	CH_3COOH	5
Zn^{2+}	$ZnCl_2$	Co-Extrn	$SO4\ _{2}^{-}$	H_2SO_4	1.5
U^{6+}	$UO_2(NO_3)_2.6H_2O$	Co-Extrn	BO_3^{3-}	H_3BO_3	2.5
Pb^{2+}	$Pb(NO_3)_2$	3	Tartrate	Tartaric acid	4
Cr^{3+}	$Cr(NO_3)_3.9H_2O$	0.3	EDTA	EDTA	1.5
Fe^{3+}	$FeCl_3.6H_2O$	1.5	Ascorbate	Ascorbic acid	2
Al^{3+}	$Al(NO_3)_3.9H_2O$	0.1			

3.5 Separation of Cadmium(II) from Multi component mixtures

Separation of Cadmium(II) was carried out from number of associated elements in multi component mixture. The mixture containing lithium(I), cadmium (II),Nickel(II), lead(II), sodium(I), potassium (I), iron(III) was passed through the poly[dibenzo-18-crown-6] column at 1×10^{-3} M glycine concentration. The lithium (I), sodium (I), potassium (I) and iron (III) was not sorbed and hence passed through the column. The cadmium (II), Nickel (II), lead (II) were sorbed. The sorbed nickel (II) was first eluted with 25 mL of 0.1 M acetic acid. After that lead (II) were eluted with 4.5 M hydrotropic acid and finally the cadium (II) was eluted with 1.0 M hydrochloric acid and effluents are analyzed spectrophotometric ally. Using this method, separation of cadmium (II), Nickel (II), lead (II) mixtures was achieved. The results are shown in the Table 5.

Table 5: Separation of Cadmium(II) from Multi component Mixtures

No	Mixture	Taken µg	Found µg	Recovery%	Sorption Condition	Eluent
1	Li(I)	50	50	100	$1x10^{-3}$M Glycine	NSPC
	Ni(II)	50	49	98		$0.1MCH_3COOH$
	Cd(II)	50	50	100		1.0 M HCl
	Pb (II)	50	50	100		4.5 M HBr
2	Na(I)	50	50	100	$1x10^{-3}$ M Glycine	NSPC
	Ni(II)	50	50	100		$0.1MCH_3COOH$
	Cd(II)	50	50	100		1.0 M HCl
	Pb (II)	50	50	100		4.5 M HBr

3	K(I)	50	50	100	1x10^{-3}M Glycine	NSPC
	Ni(II)	50	49.5	99		0.1MCH$_3$COOH
	Cd(II)	50	50	100		1.0 M HCl
	Pb(II)	50	50	100		4.5 M HBr
4	Fe(III)	50	50	100	1x10^{-3}M Glycine	NSPC
	Ni(II)	50	49	98		0.1MCH$_3$COOH
	Cd(II)	50	50	100		1.0 M HCl
	Pb(II)	50	50	100		4.5 M HBr

*NSPC = No sorption and hence passes through the column

3.6. Determination of cadmium in wood˒s metal

1.0 g woods metal was dissolved in aqua regia and evaporated almost to dryness, it was then treated with 5 ml hydrochloric acid and evaporated almost to dryness and was extracted with water and diluted to 250 ml. An aliquot of sample solution was subjected as per the above method. The results of triplicate analysis are shown below.

Table 6: Determination of cadmium in Wood˒s Metal

Alloy	Percentage of cadmium (II)	
	Present	Found
Wood ˒s Metal	10	9.98

4. Conclusion

The important feature of this method is that using column chromatographic method and poly [dibenzo-18-crown-6].The separation of Cadmium(II) from associated element in glycine medium has been achieved. The capacity of poly [dibenzo-18-crown-6] for Cadmium(II) was found to be 0.944 ± 0.01 mmol/g of crown polymer. Cadmium(II) was separated from number of cations in binary as well as multi component mixtures. The method was extended to the determination of Cadmium(II) in real sample Wood's metal. The aim of present investigation is to demonstrate a simple, selective and cheap method and has good reproducibility (approximately ± 2%).

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Sorption Study of Bismuth (III) in Glycine Medium Using Poly[dibenzo-18 crown-6] and Column Chromatography

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ABSTRACT *A simple chromatographic separation method has been developed for quantitative sorption of Bismuth(III) from an aqueous solution of* 5×10^{-3} *M Glycine using poly [dibenzo-18-crown-6] as stationary phase. The sorption of Bismuth(III) was quantitative* 1×10^{-2}*M to* 1×10^{-6}*M Glycine. The elution of Bismuth(III) was quantitative with 0.1–8.0 M HCl, 3-8M HClO4, M 1.0-8.0 M H2SO4. The capacity of poly [dibenzo-18-crown-6] for Bismuth(III) was found to be 0.306 ± 0.01 mmol/g of crown polymer. The effects of concentrations of Glycine, Bismuth(III) , foreign ions and eluents have been studied. Bismuth(III) was separated from a number of cations in multicomponent mixtures. The applicability of the proposed method was ascertained for the determination of Bismuth(III) in real samples. The reliability of method was checked by comparison of the results with those obtained using flame photometer. The method is very simple, rapid and selective with good reproducibility (approximately±2%).*

Keywords: *sorption, separation, Bismuth(III), Glycine, poly [dibenzo-18- crown-6], Chromatography*

1 Introduction

Bismuth and its compounds came to be known in the fourteenth century. However the metal at first could not be distinguished from lead and tin. It occurs in Free State as well as in the combined form. The important sources of bismuth are bismuth glance Bi_2S_2, bismuth telluride $Bi_3Te_3$and bismuth oxide (Bi_2O_3). Bismuth is found in earth's crust, up to 0.0002%. It is least toxic among the heavy metals. Bismuth has been used in the form of sub carbonates and sub galletes for the treatment of diarrhea, dysentery and ulcers. Bismuth is also used in the manufacture of low melting alloys which finds application in the fusible elements in automatic sprinklers, special solders; safety plugs in compressed gas cylinders and automatic shutoffs for gas and electric water heating systems.

Bismuth has been used in medicines for the treatment of helicobacter pylori-induced gastritis [1,2]. Bismuth and its compounds are also used in semiconductors, cos-metic preparations, alloys and metallurgical additives and in the preparation and recycling of uranium nuclear fuels [3]. As the uses of bismuth in medicine increase, bismuth has spread in the environment, and the chance of exposure of organisms to bismuth has increased [1]. Several methods have been developed for the determination of bismuth. These methods include hydride generation inductively coupled plasma atomic emission spectrometry (HG-ICPAES) [4], electro thermal vaporization ICP mass spectrometry (ETV-ICP-MS) [5], atomic absorption spectrometry (AAS) [6,7], electro thermal atomic absorption spectrometry (ETAAS) [8] potentiometric stripping analysis (PSA) [9], anodic stripping voltammetry [10,11], cathodic stripping voltammetry (CSV) [12]and amperometry [13]. However, due to the presence of low levels of bismuth in environmental samples, the separation of bismuth from other elements and the use of preconcentration are typically necessary. Conventional solvent extraction and separation of Bi in the presence of coextracting ligands, such as bis(2,4,4,-trimethylpentyl) monothiophosphinic acid [14] and pyrrolidine dithio-carbamate [15], has attracted considerable attention. The disadvantages of liquid/liquid extraction include the use of large volumes of organic solvents, cumber some glassware and cost. However, several other techniques for preconcentration of Bi have been proposed including preconcentration using Mg -W cell electrode position[16], preconcentration with sodium di-n-propyl dithiophosphinate and activated carbon in a batch method, a flow injection on-line two stage solvent extraction[17], a flow injection on-line sorption preconcentration[18–20], a fluorescence quenching method [21] and solid phase extraction [22–28].The spectrophotometric method is a sensitive technique for the determination of trace amounts of bismuth(III) in aqueous media [29–36].

2 Experimental

2.1. Apparatus and reagents

A Ziess (German) Spectrophotometer, a digital pH meter (Model LI-120, ELICO, India) with glass and calomel electrodes and a digital Flame photometer (PI, Model No. 041, India) were used. A stock solution of Bismuth (III) was prepared by dissolving 2.331 g of Bismuth (III) nitrate (Loba Chemie Pvt Ltd, Mumbai, 132, India) in 1000 mL with distilled water in a standard flask and standardized by known methods [37]. A solution containing 100 µg/mL of Bismuth(III)was prepared by appropriate dilution of standard stock solution. Glycine solution (1×10^{-1} M) was prepared by dissolving 1.875 g of glycine in distilled deionised water and diluted to 250 mL.

2.2 General procedure

40 µg of Bismuth(III) was mixed with glycine in the concentration range of 1×10^{-1}M to 1×10^{-10} M in a total volume of 10 mL. The solution was then passed through poly[dibenzo-18-crown-6] column, preconditioning with same concentration of glycine as that of the sample solution at flow rate of 0.5 mL/min. The column was then washed with the same concentration of glycine. The sorbed Bismuth(III) was then eluted with different eluting agents (described later) at the flow rate of 0.5 mL/min. 5.0 mL fraction were collected and Bismuth(III) in the aqueous phase was determined spectrophotometrically with Dithizon at 490 nm[38]. The concentration of Bismuth(III) was calculated from a calibration graph.

3. Results and Discussion

3.1. Sorption of Bismuth(III) on poly[dibenzo-18-crown-6] as a function of glycine concentration.

Sorption studies of Bismuth(III) were carried out from glycine medium. The concentration of glycine was varied from 1×10^{-1} M to 1×10^{-10}M. After sorption, the elution of Bismuth(III) was carried out with 4.0 M hydrochloric acid. It was found that there was quantitative (100%) sorption of Bismuth(III) from 1×10^{-2} M to 1×10^{-6} M L- glycine. The results are shown in **(Figure: 3.1).** The subsequent sorption studies of Bismuth(III) were carried out with 5×10^{-3} M glycine.

Table 3.1: Sorption of bismuth(III) as a function of glycine concentration Bi(III) = 40 µg, Eluent = 4.0 M HCl

Glycine concentration (M)	Sorption of Bi(III) (%)
$1x10^{-1}$	83.60
$1x10^{-2}$	100
$1x10^{-3}$	100
$1x10^{-4}$	100
$1x10^{-5}$	100
$1x10^{-6}$	100
$1x10^{-7}$	90.20
$1x10^{-8}$	87.10
$1x10^{-9}$	81.30
$1x10^{-10}$	75.12

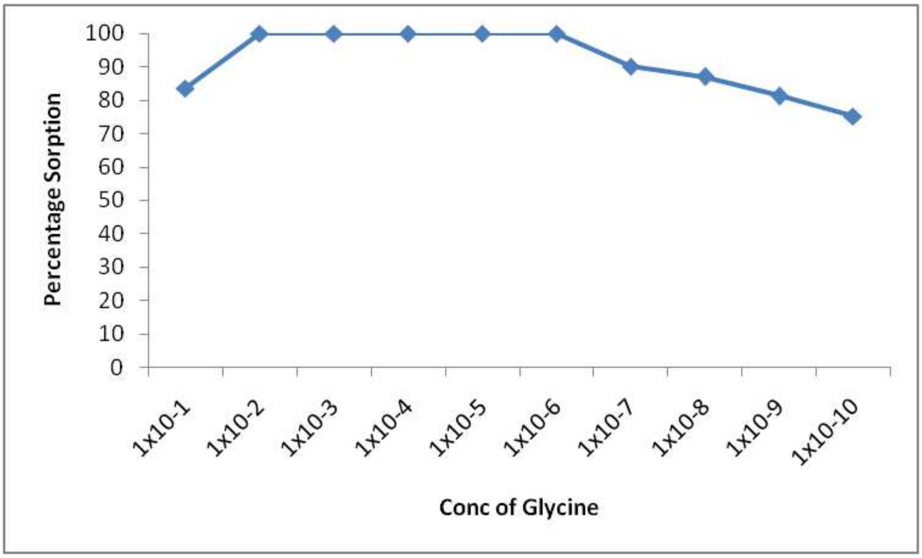

Figure: 3.1: Sorption of Bismuth(III) as a function of glycine concentration.

3.2. Elution study of Bismuth(III) with various eluting agents

40 µg/mL of Bismuth(III) was sorbed on the poly[dibenzo-18-crown-6] column at 5×10^{-4} M glycine concentration. After sorption, elution of Bismuth(III) was carried out using hydrochloric acid, sulphuric acid, perchloric acid and acetic acid. The concentration of eluting agents varied from 0.1 M to 8.0 M. The elution profile of Bismuth(III) with various eluting agents is shown in (**Figure: 3.2**). It showed that Bismuth(III) was quantitatively eluted with 0.1 M to 8.0 M hydrochloric acid, 0.1M to 8.0 M sulphuric acid ,3.0M to 8.0 M perchloric acid, Where as acetic acid was found to be inefficient eluent for Bismuth(III). Further elution studies of Bismuth(III) in this work was carried out with 4.0 M hydrochloric acid.

Table 3.2: Elution of Bismuth(III) with different eluting agents Bi(III) = 40 µg, glycine 1 x 10^{-3} M

Conc.	0.1	0.5	1.0	2.0	3.0	4.0	5.0	6.0	7.0	8.0
Acid		Percentage Elution (%)								
HCl	100	100	100	100	100	100	100	100	100	100
H_2SO_4	100	100	100	100	100	100	100	100	100	100
$HClO_4$	75.28	82.57	89.47	91.21	100	100	100	100	100	100
CH_3COOH	65.24	72.64	78.49	80.91	85.17	89.42	91.54	93.39	94.62	96.18

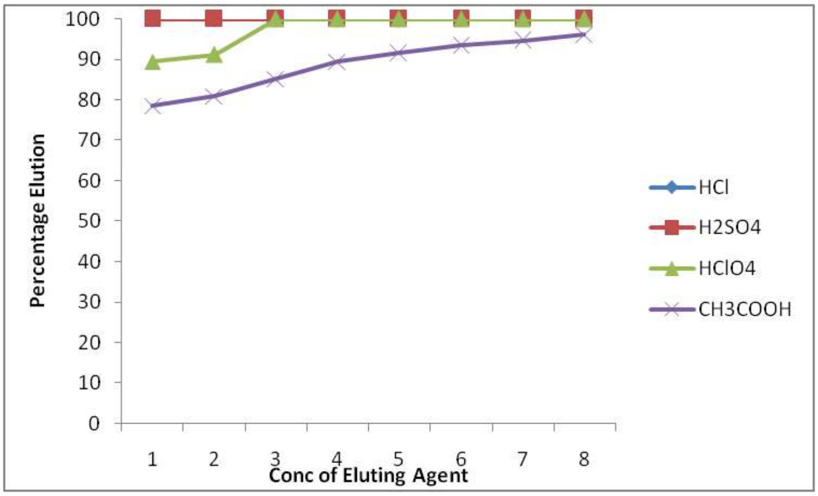

Figure: 3.2: Elution of Bismuth(III) with different eluting agents.

3.3. Effect of varying concentration of Bismuth(III)

In order to find out the capacity of poly[dibenzo-18-crown-6] for the Bismuth(III), the concentration of Bismuth(III) was varied from 40-480 µg/10 mL in glycine and 4.0 M hydrochloric acid as eluent. The results (Table 3.3) showed that the sorption of Bismuth(III) was quantitative (100%) up to 320 µg. With increase in concentration of Bismuth(III) there was decrease in the percentage sorption of Bismuth(III) and is shown in the **Figure: 3.3**. From this study it was found that the capacity of poly[dibenzo-18-crown-6] for Bismuth(III) was found to be 0.306 ± 0.01 mmol/g of crown polymer.

Table3.3. : Effect of varying concentration of Bismuth(III) glycine 1×10^{-3} M, eluent 1.0 M HCl

Bismuth(III) (µg)	Percentage sorption (%)
40	100
80	100
120	100
160	100
200	100
240	100
280	100
320	100
360	95.38
400	92.74
440	88.42
480	79.86

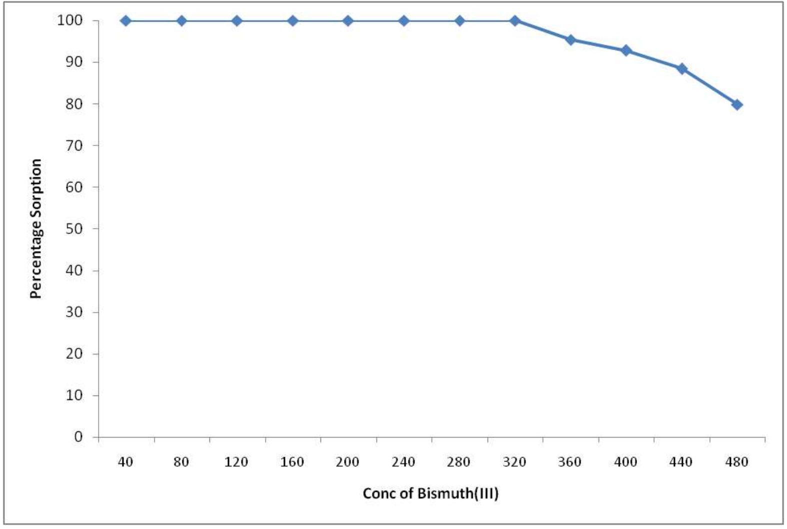

Figure: 3.3: Effect of varying concentration of Bismuth(III).

3.4 Separation of Bismuth(III) from binary mixtures

An aliquot of solution containing 40 µg of Bismuth(III) was mixed with foreign ions and glycine was added so that its concentration was 1×10^{-3} M in total volume of 10 mL. The tolerance limit was set as the amount of foreign ions required to cause ±2% deviation in the recovery of Bismuth(III). The solution was passed through a poly[dibenzo-18-crown-6] column, preconditioned with 1×10^{-3}M glycine at a flow rate of 0.5 mL/min. Subsequently the column was washed with 15 mL of 1×10^{-3}M glycine to remove unsorbed metal ions. Various foreign ions were not sorbed and hence passed through the column. The effluent was collected and analyzed for foreign ion content. The tolerance limit of various foreign ions is shown in Table **5.3.4.**

Table3.4: Separation of Bismuth(III) from binary mixtures Bi(II) = 40 µg, sorption- 1×10^{-3} M glycine, eluent- 4.0 M HCl

Ion	Added as	Tol. Limit (mg)	Ion	Added as	Tol. Limit (mg)
Li^+	LiCl	11	Tl^{3+}	$Tl(NO_3)_3.3H_2O$	0.7
Na^+	NaCl	8	La^{3+}	$La(NO_3)_3.6H_2O$	0.4
K^+	KCl	15	Ce^{3+}	$CeCl_3.6H_2O$	0.6
Rb^+	RbCl	12	V^{4+}	$VOSO_4.4H_2O$	0.3
Cs^+	CsCl	10	Th^{4+}	$Th(NO_3)_4$	Co-Extrn
NH_4^+	NH_4Cl	14	Cr^{6+}	$K_2Cr_2O_7$	0.6
Be^{2+}	$BeSO_4.4H_2O$	Co-Extrn	Mo^{6+}	$(NH_4)_6Mo_7O_{24}.4H_2O$	2.7
Mg^{2+}	$MgCl_2.6H_2O$	20	W6+	$Na_2WO_4O.4H_2O$	4
Ca^{2+}	$CaCl_2$	16	Cl^-	HCl	6
Sr^{2+}	$Sr(NO_3)_2$	8	Br-	HBr	4
Ba^{2+}	$Ba(NO_3)_2$	0.7	SCN^-	NaSCN	3
Co^{2+}	$CoCl_2.6H_2O$	Co-Extrn	ClO_4^-	$HClO_4$	2
Ni^{2+}	$NiCl_2.6H_2O$	Co-Extrn	CH_3COO^-	CH_3COOH	4
Zn^{2+}	$ZnCl_2$	Co-Extrn	SO4 $_2^-$	H_2SO_4	3.5
U^{6+}	$UO_2(NO_3)_2.6H_2O$	Co-Extrn	BO_3^{3-}	H_3BO_3	2.8
Pb^{2+}	$Pb(NO_3)_2$	2	Tartrate	Tartaric acid	5
Cr^{2+}	$Cr(NO_3)_3.9H_2O$	0.3	EDTA	EDTA	12
Fe^{3+}	$FeCl_3.6H_2O$	2.5	Ascorbate	Ascorbic acid	4

3.5 Separation of Bismuth(III) from Multi component mixtures

Separation of Bismuth(III) was carried out from number of associated elements in multi component mixture. The mixture containing lithium(I),Copper(II), Bismuth(III), sodium(I), potassium(I), thallium(III) and Manganese (II) was passed through the poly[dibenzo-18-crown-6] column at 1×10^{-3} M glycine concentration. The lithium(I), sodium(I), potassium(I) was not sorbed and hence passed through the column. The Copper(II), Bismuth(III), thallium(III) and Manganese (II) were sorbed. The sorbed Copper(II) was first eluted with 25 mL of 0.2 M LiOH. After that Bismuth(III) were eluted with 4 M hydrotropic acid and effluents are analyzed spectrophotometrically. Using this method, separation of Bismuth(III), thallium(III) and Manganese (II) mixtures was achieved. The results are shown in the **Table: 3.5.**

Table 3.5.: Separation of Bismuth(III) from Multi component Mixtures

No	Mixture	Taken µg	Found µg	Recovery %	Sorption Condition	Eluent
1	Li(I)	50	50	100	1×10^{-3}M Glycine	NSPC
	Cu(II)	50	49	98		0.2 M LiOH
	Bi(III)	40	40	100		4 M HCl
2	Na(I)	50	50	100	1×10^{-3} M Glycine	NSPC
	Cu(II)	50	50	100		0.2 M LiOH
	Bi(III)	40	40	100		4 M HCl
3	K(I)	50	50	100	1×10^{-3}M Glycine	NSPC
	Tl(III)	50	49.5	99		0.2 M A. C.
	Bi(III)	40	40	100		4 M HCl
4	Li(I)	50	50	100	1×10^{-3}M Glycine	NSPC
	Mn(II)	50	49	98		1M LiOH
	Bi(III)	40	40	100		4.5 M HCl

*NSPC = No sorption and hence passes through the column, A. C.- Ammonium carbonate

3.6. Determination of bismuth from real sample

Analysis of Bismuth(III) from Pharmaceutical sample-

The determination of Bismuth III in Denol Tablet was carried out using following procedure. The sample of Denol tablet was dissolved in 10 ml Conc. HCl. The solution was evaporated almost to dryness and sample solution was prepared. Aliquot of solution was taken from which Bismuth(III) was extracted and analyzed as per general procedure.

Sample	**Bismuth(III) mg**	
Denol Tablet	Present	Found
	75	74.30

4 Conclusion

The important feature of this method is that using column chromatographic method and poly[dibenzo-18-crown-6].The separation of Bismuth(III) from associated element in glycine medium has been achieved. The capacity of poly[dibenzo-18-crown-6] for Bismuth(III) was found to be 0.306 ± 0.01 mmol/g of crown polymer. Bismuth(III) was separated from number of cations in binary as well as multi component mixtures. The method was extended to the determination of Bismuth(III) in real sample Denol tablet. The aim of present investigation is to demonstrate a simple, selective and cheap method and has good reproducibility (approximately ± 2%).

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Sorption study of beryllium (II) in glycine medium using poly [dibenzo-18 crown-6] and column chromatography

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Abstract
A simple chromatographic separation method has been developed for quantitative sorption of Beryllium(II) from an aqueous solution of 1×10^{-3}M Glycine using poly [dibenzo-18-crown-6] as stationary phase. The sorption of Beryllium (II) was quantitative 1×10^{-3}M to 1×10^{-8}M Glycine. The elution of Beryllium (II) was quantitative with 0.1–8.0 M HCl, 3.0-8.0M HBr and 7.0-8.0 M H2SO4. The capacity of poly [dibenzo-18-crown-6] for Beryllium (II) was found to be 0.65 ± 0.01 mmol/g of crown polymer. The effects of concentrations of Glycine, Beryllium (II), foreign ions and eluents have been studied. Beryllium (II) was separated from a number of cations in multicomponent mixtures. The applicability of the proposed method was ascertained for the determination of Beryllium (II) in real samples. The reliability of method was checked by comparison of the results with those obtained using flame photometer. The method is very simple, rapid and selective with good reproducibility (approximately±2%).

Keywords: sorption, separation, Beryllium (II), Glycine, poly [dibenzo-18- crown-6], Chromatography

1. Introduction

Beryllium is industrially valuable metal due to its unique physico-chemical properties. Beryllium is rare in the earth's crust and its presence is of importance. Beryllium is particularly valuable in specific areas of nuclear technology. Beryllium is widely used in metal-alloys, salts and X-ray tubes. Due to the ability of beryllium to reflect neutrons and its efficiency in the production of neutrons, when exposed to alpha emitter has led to its use in nuclear reactors and nuclear weapons. However use of beryllium carries with it some serious health hazards, including cancer and chronic beryllium disease (CBD) [1-4].

CBD has no known cure usually and result in death. Beryllium has been linked to a number of other health effects resulting from inference of enzyme function, DNA synthesis, protein phosphorylation and cell division. An estimated 10-20 million pounds per year of beryllium are emitted during coal and refuse incineration, which can contaminate air, drinking water, soil and agricultural products [5]. Due to the high toxicity of beryllium, there is a need to separate a beryllium from other. Beryllium is one of the toxic elements, which causes some health hazards. Hence, it is necessary to analyze microgram levels concentrations of beryllium in industry and environment sample.

In the growing field of separation science numerous methods have been describe for the separation and determination of beryllium viz. Solvent extraction [6-8] ion exchange [9-11] and solid phase extraction [12-14] are among them but conventional extraction chromatography –liquid chromatography is comparatively fast, efficient and popular method.

Macrocyclic compound are uncharged and contain a cavity in which a cation can be encapsulated The complexes thus formed are of great analytical interest. It was in 1967 when Pedersen [15] published his first paper on crown ether under the title "Cylic Polyethers and their Complexes with Metal Salts." Since then these ligand have been used by physical, organic, inorganic, biochemists and also analytical chemists [16].

No attempts were made for the separation of beryllium from associated element using amino acid media and column chromatography. This chapter describes in detail the sorption study and separation of beryllium(II) using glycine required medium on poly [dibenzo-18-crown-6].The concentration of glycine required for quantitative sorption of beryllium (II) is very low, clean cut separation with good separation yield was achieved. The beryllium was successfully determined in beryl ore by using this method.

2. Experimental

2.1 Apparatus and reagents

A Ziess (German) Spectrophotometer, a digital pH meter (Model LI-120, ELICO, India) with glass and calomel electrodes and a digital Flame photometer (PI, Model No. 041, India) were used. A stock solution of beryllium(II) was prepared by dissolving 9.828 g of beryllium sulphate tetrahydrate(AR grade, BDH, Poole, England) in 100mL distilled deionised water and standardized gravimetrically [17].

A solution containing 10µg/mL of beryllium (II) was prepared by appropriate dilution of the standard stock solution. Glycine solution (1×10^{-1} M) was prepared by dissolving 1.875 g of glycine in distilled deionised water and diluted to 250 mL.

Poly [dibenzo-18-crown-6] (E Merck Darmstadt, Germany) was used after screening to 100-200 mesh. A total of 0.5 g of polymer was slurred with distilled deionised water and poured into a Pyrex glass chromatographic column (20×0.8 cm i.d.). The column was used after preconditioning with glycine solution.

2.2 General procedure

10µg of beryllium (II) was mixed with glycine the concentration range of 1×10^{-1}M to 1×10^{-11} M in a total volume of 10 mL. The solution was then passed through poly [dibenzo-18-crown-6] column, preconditioning with same concentration of glycine as that of the sample solution at flow rate of 0.5 mL/min. The column was then washed with the same concentration of glycine. The sorbed beryllium (II) was then eluted with different eluting agents (described later) at the flow rate of 0.5 mL/min. 5.0 mL fraction were collected and beryllium(II) was determined spectrophotometrically by Eriochrome cyanine R at 525nm [18] using a calibration graph.

3 Results and Discussion

3.1 Sorption of beryllium (II) on poly [dibenzo-18-crown-6] as a function of glycine concentration

Sorption studies of beryllium (II) were carried out from glycine medium. The concentration of glycine was varied from 1×10^{-1}M to 1×10^{-11} M. After sorption, the elution of beryllium (II) was carried out with 4.0 M hydrochloric acid. It was found that there was quantitative (100%) sorption of beryllium (II) from 1×10^{-3} M to 1×10^{-8} M glycine. The results are shown in (Table 1). The subsequent sorption studies of beryllium (II) were carried out with 1×10^{-5} M glycine.

Table 1: Sorption of beryllium (II) as a function of glycine concentration

Concentration of Glycine (M)	Percentage of sorption (%)
1X 10^{-1}	80.85
1X 10^{-2}	81.03
1X 10^{-3}	100
1X 10^{-4}	100
1X 10^{-5}	100
1X 10^{-6}	100
1X 10^{-7}	100
1X 10^{-8}	100
1X 10^{-9}	84.57
1X 10^{-10}	81.48
1X 10^{-11}	77.32

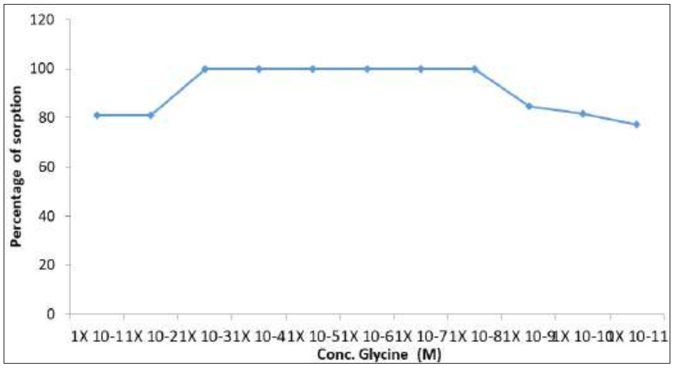

Fig 1: Sorption of beryllium (II) as a function of glycine concentration

3.2 Elution study of beryllium (II) with various eluting agents

10 µg/mL of beryllium (II) was sorbed on the poly [dibenzo-18-crown-6] column at 1×10^{-5} M glycine concentration. After sorption, elution of beryllium (II) was carried out using hydrochloric acid, hydrobromic acid, sulphuric acid, perchloric acid and acetic acid. The concentration of eluting agents varied from 0.1 M to 8.0 M. The elution profile of beryllium (II) with various eluting agents is shown in (Table 2). Showed that beryllium (II) was quantitatively eluted with 0.1 M to 8.0 M hydrochloric acid, 7.0 -8.0 M sulphuric acid and 3.0 -8.0M hydrobromic acid where as perchloric acid and acetic acid were found to be inefficient eluents for beryllium (II). Further elution studies of beryllium (II) in this work was carried out with 4.0 M hydrochloric acid.

Table 2: Elution study of beryllium (II) with various eluting agent

Conc. Acid	0.1	0.5	1.0	2.0	3.0	4.0	5.0	6.0	7.0	8.0
	Percentage of Elution (%)									
HCl	100	100	100	100	100	100	100	100	100	100
H_2SO_4	75.98	78.19	80.08	89.18	91.76	94.50	95.74	98.02	100	100
$HClO_4$	66.93	69.53	71.65	78.66	82.20	84.20	85.40	87.78	91.66	92.67
HBr	90.07	92.37	96.15	98.16	100	100	100	100	100	100
CH_3COOH	45.39	54.67	61.17	65.19	68.89	71.37	74.56	77.03	88.23	92.39

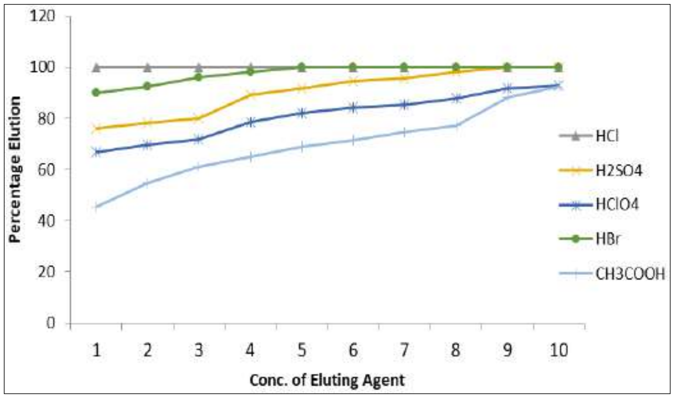

Fig 2: Elution study of beryllium (II) with various eluting agents

3.3 Effect of varying concentration of beryllium (II)

In order to find out the capacity of poly[dibenzo-18-crown-6] for the beryllium(II), the concentration of beryllium(II) was varied from 20-90 µg/10 mL in glycine and 4.0 M hydrochloric acid as eluent. The results (Table 3) showed that the sorption of beryllium(II) was quantitative (100%) up to 60 µg. With increase in concentration of beryllium(II) there was decrease in the percentage sorption of beryllium(II) and is shown in the (Figure 3) From this study it was found that the capacity of poly[dibenzo-18-crown-6] for beryllium(II) was found to be 0.65 ± 0.01 mmol/g of crown polymer.

Table 3: Effect of varying concentration of beryllium (II)

Concentration of Be(II) µg	Percentage of Sorption (%)
10	100
20	100
30	100
40	100
50	100
60	100
70	97.78
80	95.34
90	86.11

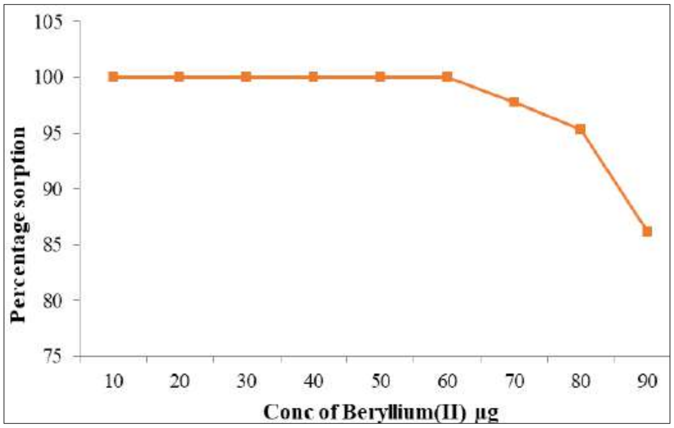

Fig 3: Effect of varying concentration of beryllium (II)

3.4 Separation of beryllium (II) from binary mixtures

An aliquot of solution containing 10 µg of beryllium (II) was mixed with foreign ions and glycine was added so that its concentration was 1×10^{-5}M in total volume of 10 mL. The tolerance limit was set as the amount of foreign ions required to cause ±2% deviation in the recovery of beryllium (II). The solution was passed through a poly [dibenzo-18-crown-6] column, preconditioned with 1×10^{-5} M glycine at a flow rate of 0.5 mL/min. Subsequently the column was washed with 15 mL of 1×10^{-5} M glycine to remove unsorbed metal ions. Various foreign ions were not sorbed and hence passed through the column. The effluent was collected and analyzed for foreign ion content. The tolerance limit of various foreign ions is shown in Table 4. The most of the alkali metals and alkaline earth metals show high tolerance limit except barium (II) and magnesium (II). In case of alkaline metal sodium (I) potassium (I) and alkaline earth metals calcium (II), strontium (II) tolerates strongly. Most of the p-block and d-block elements were sorbed and shows low tolerance limit but tungsten (VI) and lead (II) highest tolerance limit. Amongst the inner transition elements, As compared lanthanum (III), cerium (III), gadolinium (II) low tolerance limit. The anion of inorganic and organic acids showed high tolerance limit

Table 4

Ion	Added as	Tolerance limit(mg)	Ion	Added as	Tolerance limit(mg)
Li^+	LiCl	1	Th^{4+}	$Th(NO_3)_4.6H_2O$	0.05
Na^+	NaCl	7	Sb^{3+}	$SbCl_3$	0.05
K^+	KCl	6	Sn^{2+}	SnCl2 2 H2O	1
Cs^+	CsCl	1	Ce^{3+}	$CeCl_3\ 6H_2O$	0.5
Cr^{3+}	$Cr(NO_3)_3,9H_2O$	0.05	Gd3+	Gd(NO3)3, 6H2O	0.01
Mg^{2+}	$MgCl_2.6H_2O$	0.01	W^{6+}	$Na_2WO_4,4H_2O$	5
Ca^{2+}	$CaCl_2$	20	Zr^{4+}	$Zr(NO_3)_{4,}\ 4H_2O$	0.5
Sr^{2+}	$Sr(NO_3)_2$	18	La^{3+}	$La(NO_3)_3$	1
Ba^{2+}	$Ba(NO_3)_2$	0.01	NH_4^+	NH_4OH	3
Co^{2+}	$CoCl_2.6H_2O$	0.05	Citrate	Citric acid	8
Ni^{2+}	$NiCl_2.6H_2O$	0.01	CH_3COO^-	CH_3COOH	3
Mn^{2+}	$MnCl_2.4H_2O$	0.05	SO_4^{2-}	H_2SO_4	0.01
Zn^{2+}	$ZnCl_2$	0.1	Tartarate	Tartaric acid	4
Cd^{2+}	$(CH_3COO)_2\ CdH_2O$	0.05	EDTA	EDTA	0.05
Pb^{2+}	$Pb(NO_3)_2$	7	Ascorbate	Ascorbic acid	5
Fe^{3+}	$FeCl_3.6H_2O$	0.1	ClO_4^-	$HClO_4$	0.1
Al^{3+}	$AlCl_3.16H_2O$	<0.01	SCN^-	NaSCN	23
Cu^{2+}	CuCl2	0.05	Cl^-	HCl	0.1
Tl^{3+}	$Tl(NO_3)_3.3H_2O$	0.05	PO_4^{3-}	H_3PO_4	18
Mo^{6+}	$(NH_4)_6Mo_7O_{24}.4H_2O$	0.01	Bo_3^{3-}	H_3BO_3	0.5

3.5 Separation of beryllium (II) from multicomponent mixtures

Separation of beryllium (II) was carried out from number of associated elements in multicomponent mixture. The mixture containing Lithium(I), Copper(II), Lead(II), Cadmium(II), Nickel(II), Zinc (II) was passed through the poly[dibenzo-18-crown-6] column at 1×10^{-5} M glycine concentration, lithium(II) was not sorbed and hence passed through the column. The Copper (II), Lead (II), Cadmium (II), Nickel (II), Zinc (II) were sorbed. The sorbed Copper (II), was first eluted with 25 mL of 0.5 M LiOH. After that beryllium (II) were eluted with 4.0 M hydrochloric acid and effluents are analyzed spectrophotometrically. Using this method, separation of Lead (II), Cadmium (II), Nickel (II), Zinc(II) mixtures was achieved. The results are shown in (Table: 5).

Table 5: Separation of beryllium (II) from other elements (multicomponent mixture).

No.	Mixture	Taken µg	Recovery* %	Eluent
1	Li(I)	50	99.00	NSPC**
	Be(II)	10	99.5	4.0 M HCl
	Cu(II)	40	99.50	0.5 M LiOH
2	Li(I)	50	99.20	NSPC**
	Pb(II)	40	99.5	0.2M A.C
	Be(II)	10	98.5	4.0 M HCl
3	Li(I)	50	99.50	NSPC**
	CdII)	40	99.0	0.5M LiOH
	Be(II)	10	99.5	4.0 M HCl
4	Li(I)	50	99.50	NSPC**
	NiII)	40	98.5	0.2M LiOH
	Be(II)	10	99.5	4.0 M HCl
5	Li(I)	50	99.50	NSPC**
	Zn(II)	40	97.00	0.2M LiOH
	Be(II)	10	99.5	4.0 M HCl

*Average of triplicate analysis

**NSPC- No Sorption Passing through the Column, A.C.-Ammonium carbonate

3.6 Determination of beryllium (II) in beryl ore sample

A finely powdered 0.5 g of beryl ore sample was fused with a mixture of sodium hydroxide and sodium peroxide. The cooled mass was loosened with water. The precipitated hydroxides of beryllium, magnesium and iron were digested on water bath and later filtered. The precipitated mass was dissolved in dilute hydrochloric acid and diluted to 250 mL [19]. An aliquot of sample solution was taken and subjected to proposed method. The separated beryllium (II) content was analyzed by spectrophotometer using Eriochrome cyanine-R indicator. The amount of beryllium (II) found by triplicate determination is 13.35 % as against the reported value of 13.4%.

4. Conclusion

The important feature of this method is that using column chromatographic method and poly [dibenzo-18-crown-6] the separation of beryllium (II) from associated element in glycine medium has been achieved. The capacity of poly [dibenzo-18-crown-6] for beryllium (II) was found to be 0.65 ± 0.01 mmol/g of crown polymer. Beryllium (II) was separated from number of cations in binary as well as multicomponent mixtures. The method was extended to the determination of beryllium in real sample. The aim of present investigation is to demonstrate a simple, selective and cheap method and has good reproducibility (approximately ± 2%).

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Column chromatographic separation of uranium (vi) and other elements using Poly(dibenzo-18-crown-6) and glycine medium

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Proceedings of the fourteenth biennial DAE-BRNS symposium on nuclear and radiochemistry: book of abstracts

Abstract

[en] A simple and efficient column chromatographic method has been developed for the separation uranium (VI) and Thorium (IV) and other metal using poly(dibenzo-18-crown-6) as stationary phase and glycine as counter ion. The various eluting agents were found efficient eluents for uranium(VI). The capacity of crown polymer for uranium (VI) was found to be 1.92±0.01 mmol g”1. The tolerance limit of various cations and anion for uranium (VI) was determined. Uranium (VI) was qualitatively separated from other metal ion in binary as well as multiple component mixtures. The good separation yield were obtained and had good reproducibility (±2%). The method incorporated the determination of uranium in geological samples and in some water sample around Kolhapur City. The method was simple, rapid and selective

Primary Subject

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(57) Abstract :

ABSTRACT Biological specimen preservation by potash alum crystal In the given invention, Biological specimen preservation by potash alum crystal • the simple, economical method for biological specimen is given. In this method the interested specimen (1) of the rare and endangered species is initially coated with the coating of silver or similar base nano particles (2) which forms protective covering on the specimen and protect it from moisture and dust. After properly coating the specimen , the coated specimen is enclosed into the potash alum octahedral crystal by crystallization method i.e. coated specimen is inserted into saturated solution of the potash alum and then solution is allowed to evaporate , when the saturated solution evaporates completely then the specimen will get enclosed into octahedral shape crystal(3) of the potash alum. Depending upon the required size of the crystal quantity of saturated solution is used. Then this potash alum octahedral crystal containing specimen is also coated with the silver or similar base nano particles , which forms protective covering(4) for the potash alum crystal for protecting it from water , dust & other environmental factors.

No. of Pages : 16 No. of Claims : 3

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1. APPLICANT(S):

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3. TITLE OF THE INVENTION: Biological specimen preservation by potash alum crystal

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5. PRIORITY PARTICULARS OF THE APPLICATION(S) FILED IN CONVENTION COUNTRY:

Sr.No.	Country	Application Number	Filing Date	Name of the Applicant	Tilte of the Invention

6. PARTICULARS FOR FILING PATENT COOPERATION TREATY (PCT) NATIONAL PHASE APPLICATION:

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7. PARTICULARS FOR FILING DIVISIONAL APPLICATION

Original (first) Application Number	Date of Filing of Original (first) Application

8. PARTICULARS FOR FILING PATENT OF ADDITION:

Main Application / Patent Number:	Date of Filing of Main Application

9. DECLARATIONS:

(i) Declaration by the inventor(s)

I/We ,Miss Patil Pratibha Suresh , is/are the true & first inventor(s) for this invention and declare that the applicant(s) herein is/are my/our assignee or legal representative.

(a) Date: -----

(b) Signature(s) of the inventor(s): ....

(c) Name(s): Miss Patil Pratibha Suresh

(ii) Declaration by the applicant(s) in the convention country

I/We, the applicant(s) in the convention country declare that the applicant(s) herein is/are my/our assignee or legal representative.

(a) Date: -----

(b) Signature(s) : ....

(c) Name(s) of the singnatory: Dr. Nikam Nitin Dattatray

(iii) Declaration by the applicant(s)

- **The Complete specification relationg to the invention is filed with this application.**
- **I am/We are, in the possession of the above mentioned invention.**
- **There is no lawful ground of objection to the grant of the Patent to me/us.**

10. FOLLOWING ARE THE ATTACHMENTS WITH THE APPLICATION:

Sr.	Document Description	FileName
1	COMPLETE SPECIFICATION	Complete Specifications.pdf
2	DRAWINGS	drawing details.pdf

I/We hereby declare that to the best of my/our knowledge, information and belief the fact and matters stated hering are correct and I/We request that a patent may be granted to me/us for the said invention.

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