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FINAL REPORT
INFLIGHT URINE VOLUME DETERMINATION

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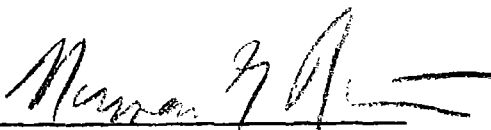
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URINE VOLUME DETERMINATION STUDY

1.0 OBJECTIVE:

The objective of this work is to conduct experiments to determine the adequacy of a chemical tracer method for the determination of total urine volumes excreted during space flight.

2.0 INTRODUCTION:

The method defined is based on the use of lithium or cesium salts as tracer materials, and atomic absorption spectrophotometry as an analytical method for the determination of the tracer. In this method, the urine container is charged with a known quantity of salt, the urine is allowed to mix with the salt, a sample of the mixture is collected for analysis, and the remainder may then be jetisoned overboard. A portion of the collected sample is analyzed for lithium or cesium concentration, thereby allowing a determination of urine void excreted in flight from which the sample was withdrawn.

Since the literature related to the analysis of urine has not indicated the presence of either lithium or cesium, these elements would appear to be most suitable for the subject analysis. Both elements may be readily analyzed in trace quantities by atomic absorption spectrophotometry, the salts are readily soluble, and in small quantities should not interfere with normal urinalysis procedures.

This project was initiated to investigate the feasibility, functionality, and problem areas involved in using lithium and cesium as tracers for urine volume determinations. Experiments were

performed to: determine the presence of lithium or cesium in urine, or the possible interference of urine with the analysis of the tracers, determine the rates of dispersion of the tracer salts in urine, determine the possible retention of the tracer by the urine collection bag materials, and to determine the overall accuracy and functionality of the method. In addition, the literature was searched to determine if other investigators have detected the presence of either lithium or cesium in urine.

3.0

DISCUSSION AND RESULTS

3.1

Literature Search

The technical literature was searched to determine if others have reported detecting the presence of either lithium or cesium in normal urine.

This search revealed no reference to the presence of lithium. There were a few references to studies involving the detection of radioactive cesium in urine; however, these studies were related to detecting extremely low levels of Cs-137 by scintillation counting methods, and at levels far below those that would interfere with cesium analysis by atomic absorption spectrophotometry.

In light of the findings of our laboratory experiments related to the interference levels of urine to the analysis of cesium, we now consider cesium a poor candidate as a tracer; and, therefore, have elected to abandon further searching beyond those sources

cited below. The following were the major sources searched:

1. Documentation Geigy - Science Tables, 6th ed. Pub. Geigy Pharmaceuticals.
2. Biology Data Book for Federation of America Society for Experimental Biology - Washington D.C., Compiled and Edited by Philip L. Altman and D.S. Dittmer.
3. International Critical Tables of Numerical Data Physics, Chemistry and Technology, Published for the National Research Council by the McGraw-Hill Book Company, Inc., New York and London 1933.
4. NASA Life Sciences Data Book 1962, Compiled by Webb Associates, Yellow Springs, Ohio, NASA Publication SP-3006.
5. Biological Abstracts 1967-1969.

3.2

Determination of the Presence of Lithium and Cesium in Urine or Interference Contributed by Urine in the Analysis of Lithium and Cesium by Atomic Absorption Spectrophotometry.

3.2.1

Procedure

Urine samples were obtained from ten (10) male subjects and analyzed for lithium and cesium by atomic absorption spectrophotometry. A Perkin-Elmer Model 403 Spectrophotometer was used for analysis. The concentrations shown in all experiments in this report were the average of 100 samplings, obtainable with this model spectrophotometer. Standard solutions of the instrument were obtained by dilution of standard spectrographic solutions.

3.2.2

Results

The results of these tests (listed in Table 3.2.2.A) show interference caused by urine to be of sufficiently low levels in the lithium analysis to consider lithium a good choice as a tracer. All urine interference may be eliminated by dilution of the urine prior to analysis.

The interference caused by urine in the analysis of cesium was considered too high for the intended purpose as a tracer; and, therefore, cesium was eliminated from all subsequent experiments.

Lithium was analyzed by absorption. Cesium was analyzed by flame emission, since emission measurements are sensitive to this element.

The response to lithium in the samples varied from .01 to .03 ppm. The response to cesium varied from 5.0 to 9.0 ppm.

Samples of the urine specimens were diluted 10:1 with water to determine if dilution would eliminate interferences, caused by the urine, in the lithium analysis. Dilution was found to eliminate the interference on all samples. These results are also listed in Table 3.2.2.A.

The interference response to cesium in normal urine was quite significant; consequently, no attempt was made to dilute the samples for analysis since this remedy was considered inadequate to solve the problem.

Flame photometry reference literature indicates that the major

Table 3.2.2.A

Presence of Lithium and Cesium in Urine or Interference Contributed
by Urine in the Analysis of Lithium and Cesium by Atomic Absorption
Spectrophotometry

CONCENTRATION OR INTERFERENCE - PPM			
<u>Subject No.</u>	<u>Li</u>	<u>Li (Diluted 10:1)</u>	<u>Cs</u>
1	.01	0	5.0
2	.01	0	7.5
3	.01	0	5.5
4	.03	0	6.8
5	.03	0	6.5
6	.02	0	9.0
7	.03	0	7.0
8	.03	0	6.8
9	.02	0	7.0
10	.02	0	5.5

contributors to interference in lithium analysis are probably potassium, sodium, and calcium. In the cesium analysis the element believed to be contributing the major interference effects is potassium. A filter was placed in the optical system to prevent sodium interference in the cesium analysis. This did not resolve the problem.

3.3

Tracer Method Accuracy (For Lithium)

Some of the variables that may affect the analysis of the lithium tracer are listed below:

(a) Instrument Accuracy	$\pm 1-2\%$ (may vary with tracer concentration)
(b) Pipettes (NBS)	$\pm 0.3\%$ (for 2 ml.) $\pm 0.2\%$ (for 5 ml.) $\pm 0.1\%$ (for 10 ml.)
(c) Urine Interference	.01 to .03 ppm. $\pm 0\%$ (diluted urine)
(d) Urine Void Volume	(65 ml. to 580 ml.)
(e) Lithium Salt Purity	(to spectrographic standards)
(f) Urine Sample Temperature	(Spacecraft and laboratory control)
(g) Barometric Pressure	(Spacecraft and laboratory control)
(h) Tracer Adsorption or Absorption on Urine Collection Bag	(Results of sec. 3.5 show no definite interaction)
(i) Mixing of Tracer with Urine	(Some minimum degree of mixing required. Actual values unknown)
(j) Tracer Concentration	(Results of sec. 3.6 indicate that high concentration may reduce error)

Although there are several variables that may introduce errors in analysis, most of the potential variables may be checked in the laboratory against suitable standards; and may, therefore, be considered non-contributory to total accuracy. The pipettes or similar volumetric measuring equipment used for impregnating the urine collection bags, or extracting a urine sample for analysis may be suitably evaluated for accuracy against known standards. The effects of urine on analysis of lithium may be eliminated by dilution. Tracer salt purity may be checked against standard analytical solutions used to calibrate the spectrophotometer, and may, therefore, be considered to contribute no error to the method. Since temperature and pressure affect volumetric measurements, these will need to be measured or controlled within narrow limits to minimize error. Assuming adequate control or compensation of these parameters, essentially no error should be assumed in the analysis.

The amount of tracer recommended at this time is determined primarily by the accuracy and detection limits of the spectrophotometer. In order to minimize error, and also minimize the amount of tracer material required, it appears that the procedure should include a quantity of tracer sufficient to result in final analytical concentration of at least 1 ppm. lithium. In order to eliminate interference caused by urine, if we assume that the urine sample must be diluted 10:1 for analysis, the concentration in urine should be at least 10 ppm. lithium. This would result in a requirement for at least 30.7 mg. LiCl salt for a 500 ml. void

3.4

(continued)

the analysis of cesium, cesium was not included as a tracer in the dispersion experiments. Discussion of the procedures and results of the tests follows:

3.4.1

Procedure A - Dispersion from Top to Bottom of Column

Approximately 400 ml. urine was contained in a 500 ml. column (graduated cylinder type column). The spectrophotometer aspirating sampling tube was inserted to the bottom of the urine column, and secured in place to prevent movement of the tube. Five ml. of a 1000 ppm. Li (chloride) solution was carefully pipetted on to the surface of the urine, and sampling was run on a continuous basis to the spectrophotometer. The chloride was used in all of the experiments, in preference to the nitrate, since chlorides were already present in urine at high concentrations; and the tracer salt would contribute little to the total chloride content. The tracer was obtained by dilution of standard certified spectrographic solutions. The average of 100 samplings were read at intervals up to one hour, sampling was discontinued for one hour, a reading was taken at two hours, the urine solution was thoroughly mixed by capping the column and shaking for one minute. Another sample was then taken to determine equilibrium concentration.

3.4.1.1

Results

The results of this experiment (Table 3.4.1.1.A) show that dispersion was incomplete in a two hour period.

Table 3.4.1.1.A

DISPERSION FROM TOP TO BOTTOM OF COLUMN

<u>Time (mins.)</u>	<u>Li Conc., ppm.</u>
2	.02
5	.02
7	.05
10	.07
15	.10
20	.12
30	.22
40	5.02
48	4.46
56	6.56
60	6.54
Sampling Off For 1 Hr.	
120	11.3
(Mix Sample)	
124	12.69

400 mls. urine in 500 ml. column

3.4.1.1 (continued)

The sampling rate required by the spectrophotometer was found to be approximately 0.5 ml./min. Therefore, approximately 30 ml. of solution was extracted, by sampling, during the first hour of the test.

3.4.2 Procedure B - Dispersion From Bottom to Top of Column

Two tests were run to determine dispersion rate of the tracer from the bottom to the top of a urine column. These tests are described below.

3.4.2.1 Procedure - Test A

One hundred mls. of urine were contained in a 100 ml. graduated cylinder. Approximately 1 ml. of a 100 ppm. Li (Cl) solution was inserted to the bottom of the urine column with a pipette, and the pipette was slowly withdrawn. Sampling was run continuously, with the sampling tube located approximately 1/3 of the distance down from the top of the solution. Sampling was continued for one hour, the solution was then shaken for one minute, and a final reading taken.

3.4.2.1.1 Results

The results are shown on Table 3.4.2.1.A and indicate that dispersion was incomplete after one hour.

Because the procedure, urine volume, and technique used in this test were considered unsatisfactory, a second experiment was performed which was much better controlled and provided more significant data on the dispersion rate. (Test B).

Table 3.4.2.1.A

DISPERSION FROM BOTTOM TO TOP OF COLUMN

Test A*		Test B**	
Time (mins.)	Li Conc., ppm.	Time (mins.)	Li Conc., ppm.
2	.58	10	0
4	.60	20	0
6	.95	30	0
7	.80	45	.01
9	.75	90	.02
11	.72	120	.02
13	.70	16 hrs.	.36
15	.71	(Mix Sample)	
25	.72	16 hrs.	1.76
30	.73		1.76
45	.72	Samples from	1.75
60	.79	Several Locations	1.77
(Mix Sample)		In Column	1.75
70	.50		1.77
			1.75

* Test A - 100 mls. urine in 100 ml. column

** Test B - 500 mls. urine (diluted 10:1) in 500 ml. column

3.4.2.2 Procedure - Test B

Considerable difficulty had been experienced in previous tests with the plugging of the spectrophotometer sampling tubing by urine. Since the dispersion tests should not be disturbed by repeatedly extracting and inserting the tube into the solution, it was decided to dilute the urine 10:1 with distilled water. Diluted urine, therefore, was used in Test B.

Approximately 500 mls. of diluted urine were contained in a cylindrical gas bottle. The gas bottle was equipped with a stopcock at each end. The bottle was positioned vertically with the upper stopcock open, and the instrument sampling tube inserted approximately 1 inch into the top of the solution through the upper stopcock. A small rubber bulb was filled with approximately 2 ml. of 1000 ppm. Li (Cl) solution and attached to the bottom stopcock. The stopcock was opened, the rubber bulb was carefully and slowly squeezed to dispense the tracer to the bottom of the urine solution, and the stopcock was then closed.

Sampling occurred continuously for the first 45 minutes, and re-started for subsequent readings at 1-1/2, 2, and 16 hours elapsed time. Table 3.4.2.1.A shows the tracer concentrations for this time period. After the 16 hour reading, the solution was thoroughly shaken for approximately two minutes, and the sampling tube moved to several locations in the solution to determine variations.

3.4.2.2.1 Results

The results, listed in Table 3.4.2.1.A, show that complete dispersion did not occur within 16 hours. After shaking, the total variation between samplings at different locations in the solution was 0.02 ppm. or ranged from 1.75 to 1.77 ppm. concentration.

3.4.3

Procedure C - Dispersion in Urine Collection Bag *

Five mls. of a 1000 ppm. Li (Cl) solution was pipetted into a urine collection bag, and the solution air-dried overnight. Approximately 500 mls. of urine was poured into the bag. With the sampling tube inserted to one corner of the bag, concentration readings were taken for two hours. The solution was then mixed for thirty seconds, samples taken from several locations in the bag; again, mixed for thirty seconds and sampled from several locations in the bag.

3.4.3.1

Results

The results, shown in Table 3.4.3.1.A indicate that dispersion was incomplete after one hour. After one 30 second mixing at two hours there was approximately 8 percent variation in readings from several locations in the bag.

It will be noted that because of a misunderstanding on the use of the collection bags, the above experiment was performed on the side of the bag containing polyurethane foam; and, therefore, may have influenced the results of the test. However, in light of the results of the experiments described in sections 3.4.1 and 3.4.2, which employed urine columns, it is reasonable to assume that, in the absence of mixing, complete dispersion would occur at a comparable slow rate in the zipper-enclosed side of the bag.

An important unknown factor that must be considered, of course, is the degree of mixing (agitation) that will occur under actual in-flight conditions. If this mixing is substantial, the dispersion rate should be considerably improved.

*Urine collection bags used in all tests were GFP, manufactured by Fairchild Hiller as their part no. RA-083C0010.

Table 3.4.3.1.A

DISPERSION OF LiCl IN URINE COLLECTION BAG

<u>Time (mins.)</u>		<u>Li Conc., ppm.</u>
10		4.47
15		4.86
25		4.29
30		4.46
35		4.45
40		4.56
45		4.52
50		4.57
55		4.66
60		4.71
120		7.6
(Mix Sample 30 sec.)		
123		7.16
125		7.24
130	Samples from Several	7.38
135	Locations in Bag.	7.54
140	Variation 0.59 ppm	7.08
145	or Approx. 8% of	7.49
150	Conc.	7.67
155		7.61
(Mix Sample 30 sec.)		
160		7.66

3.5 Adsorption, Absorption, or Reaction of Tracer with Urine Collection

Bag Materials

3.5.1 Procedure

Five ml of a 1000 ppm. Li (Cl) solution were pipetted into the polyurethane foam side of each of five urine collection bags, and the solution was air-dried for two days prior to the first test. At intervals of one week, starting with week 0, and extending to week 4, the polyethylene on the face of the bag was cut to the corners with scissors to completely expose all surfaces of the bag. The bag was then placed in a 12 inch diameter glass container. Five hundred mls. of urine, measured at 20°C with a volumetric flask, was poured into the container and mixed for 10 minutes by manually agitating the container. Ten mls. of mixed solution was withdrawn from the container with a 10 ml. pipette, placed in a 100 ml. volumetric flask, diluted to 100 mls. with distilled water, mixed thoroughly, and analyzed by inserting the spectrophotometer sampling tube into the flask.

The same pipettes and volumetric flasks were used for all tests to hold these variables constant.

3.5.2 Results

The results of these tests are listed in Table 3.5.2.A.

The lithium concentration for all of the tests should have been 1.00 ppm. The readings at 0, 2, and 3 weeks were within the limits of error anticipated for the instrument ($\pm 1-2\%$). The reading at 1 week resulted in a 6% error; and at 4 weeks, a 5% error.

Table 3.5.2.A

Adsorption, Absorption, or Reaction of Tracer with Urine Collection

Bag Materials

(Tracer and Urine in Polyurethane Foam Side of Bags)

<u>Time (Weeks</u>	<u>Lithium Conc., ppm.</u>
0	.99
1	.94
	.94 - 2 minutes additional mix
2	1.00
3	.98
4	.95
	.95 - 5 minutes additional mix

3.5.2

(continued)

Because the pattern of tracer concentration over the 4 week period indicates no definite trend, and considering the errors experienced in the series of experiments in section 3.6, it would appear that the variation in results may be attributed more to deficiencies in the technique than to interaction of the tracer with the bag.

3.6

Overall Accuracy and Functionality of the Method

Experiments were performed to determine the overall accuracy and functionality of the tracer method. The tests involved impregnating urine collection bags with lithium tracer, and drying the tracer salt in the bags. Urine volumes of 100, 250, and 500 mls. were added to different bags. The urine-tracer mixture was analyzed for lithium concentration, and urine volumes were calculated from the analyses.

Two sets of experiments were performed: one using the polyurethane foam side of the bags; and, after discussion with the contract monitor, another using the zipper-access side of the bag. Since the tests involving the polyurethane foam side of the bags were somewhat invalid, because of that side not normally being used for urine collection, the data are shown only to indicate the levels of accuracy obtained and to show the degree of mixing required to effect dispersion of the tracer.

3.6.1

Procedure - Test A

Five mls. of a 1000 ppm. Li (Cl) solution was pipetted into the

3.6.1

(continued)

polyurethane foam side of each of 5 urine collection bags, and dried overnight in an oven at 100°C. A volume of urine was added to each bag, mixing was accomplished by shaking, and 10 mls. of solution was withdrawn with a pipette. This was placed in a 100 ml volumetric flask, diluted to 100 mls. with distilled water, mixed, and analyzed.

Volumes of 100, 250, and 500 mls. urine were used for the tests.

Large, bored rubber stoppers were attached to the adhesive of the bag openings for adding urine to the bag, and a small stopper was inserted into the hole of the large stopper to effect a seal for mixing.

3.6.1.1

Results

Results of these tests are shown in Table 3.6.1.1.A.

The first test, using 100 mls. of urine, and one minute mixing times at 5 and 25 minutes elapsed time, showed a 5% error in analysis.

This test was then repeated, with longer mixing times, for a total elapsed time of 60 minutes. Under these conditions the tracer concentration achieved its proper level resulting in a 0% error.

The 250 and 500 ml tests showed errors of 14% and 4%, respectively, using the degrees of mixing and elapsed times shown in Table 3.6.1.1.A.

In order to determine if the large error in the 250 ml. test could have been caused by insufficient mixing, this test was repeated, except that the bag containing the urine was mixed on a mechanical

3.6.1.1 (continued)

shaker for 30 minutes prior to analysis. The error was thereby reduced from 14% to 3%.

Table 3.6.1.1.A

Overall Accuracy and Functionality Test A, Various Urine Volumes
in Urine Collection Bags
 (Tracer and Urine in Polyurethane Foam Side of Bags)

Urine Vol. ml.	Time from Start Minutes	Mixing Time Minutes	Li Conc., ppm.	<u>Error</u> % ml.		Urine Vol. by Analysis ml.
100	5	1	4.77			
	25	1	5.24	5	5	95
100	12	10	4.75			
	30	5	4.89			
	60	5	5.00	0	0	100
250	8	6	2.19			
	30	6	2.30			
	60	6	2.34	14	36	214
500	7	2	.90			
	20	2	.92			
	35	2	.95			
	50	2	.96	4	20	520
250	40	30 (on mechanical shaker)	1.94	3	8	258

3.6.2

Procedure - Test B

The procedure for this set of experiments was the same as that described for Test A, 3.6.1, except that the zipper-access side of the bag was used, and the quantity of urine-tracer solution withdrawn for analysis was 1 ml., instead of 10 mls. as used in section 3.6.1.

The 1 ml. sample was withdrawn from the mixed solution with a 1 ml. pipette, and placed in a small vial. To this, 10 mls. of distilled water was added with a pipette. This smaller sample quantity was used to more closely simulate actual-use conditions of analysis.

3.6.2.1

Results

The results of the tests are listed in Table 3.6.2.1.A.

The errors imposed by the method in these tests were 1 to 3%. Since the 250 and 500 ml results showed a 3% error, the solutions were re-mixed by shaking, and another 1 ml. sample extracted, diluted, and analyzed again as a re-check on the first reading; however, no change in concentration occurred by re-mixing.

It will be noted, that the tracer concentrations in Test B (this section) varied from those of Test A, section 3.6.1, since the dilutions are different. The dilutions in Test A were 9 water and 1 solution, while those in Test B were 10 water and 1 solution. The effect of the variation in dilution was included in the calculation of the results as shown.

The results of these final tests show that lithium may be used as a tracer for urine volume determinations. It is believed by

3.6.2.1 (continued)

the investigators that through a more extensive program, involving a greater number of samples and repeated use of the method, that the volume errors could be reduced. Since this program involved a somewhat limited number of tests, the influence of all variables that may contribute to error has not been thoroughly evaluated. Improvements might be realized, for instance, by use of higher concentrations of tracer, better mixing, or by attempting to reduce instrument error by improved technique.

Table 3.6.2.1.A

Overall Accuracy and Functionality Test B, Various Urine
Volumes in Urine Collection Bags
(Tracer and Urine in Zipper Side of Bags)

Urine Vol. ml.	Time from Start Minutes	Mixing Time Minutes	Li Conc., ppm.	<u>Error</u> % ml.		Urine Vol. by Analysis ml.
100	12	10	4.50	1	1	101
250	8	5	1.88			
	30	5	1.88	3	8	242
500	10	5	.94			
	30	5	.94	3	16	484

3.7 Determination of Urine Interference in the Analysis of Chromium
and Strontium

Because urine interference in the analysis of cesium (section 3.2) was found to be high, urine samples were analyzed for chromium and

3.7

(continued)

strontium, since these elements appeared to be suitable candidates for consideration as tracers in future studies.

Chromium was determined by atomic absorption, and strontium was determined by flame emission.

3.7.1

Results

The results show a response to chromium of 0.02 ppm, and to strontium of 0.18 ppm. The response to chromium appeared to be comparable to that of lithium. Therefore, chromium might be considered a candidate as an alternate or supplement tracer to lithium in future studies.

The response to strontium was higher than that to chromium. However, it may be possible in future studies to reduce the interference to lower acceptable levels through use of optical filters or similar modified instrumental technique. Strontium may, therefore, provide another alternative to the use of lithium, in the event that this is desirable in the future.

4.0

SUMMARY

4.1

Literature Search

A search of the literature revealed no reference to the presence of lithium in urine. The only references to the presence of cesium involved studies of detection of extremely low levels of radioactive cesium-137.

4.2

Interference of Urine in Analysis of Tracer

Urine specimens obtained from ten male subjects were analyzed for lithium and cesium by atomic absorption spectrophotometry. The response to lithium ranged from .01 to .03 ppm. This interference was reduced to zero response by 10:1 dilution with water.

The instrument response to cesium ranged from 5.0 to 9.0 ppm. This was believed to be primarily a response to potassium contained in the urine.

4.3

Method Accuracy

The accuracy of the method appeared to be limited primarily by the instrumentation and degree of mixing of the tracer with urine, and perhaps by the level of tracer concentration. Assuming good control over the mechanics involved in the use of the method, the instrument appeared to be the major limitation. This should have affected accuracy by no more than 1 to 2%. Higher tracer concentrations and improved technique could reduce this level of error in future determinations.

4.4

Dispersion of Tracer in Urine

Experiments on dispersion of lithium chloride in urine showed that dispersion, in the absence of mixing, was incomplete in two hours in two tests, and in 16 hours in another test. These results indicated the necessity for some degree of supplemental mixing of the urine-tracer solution prior to extracting a sample for analysis.

4.5

Interaction of Tracer with Urine Collection Bag

Experiments to determine possible interaction of the tracer with urine collection bag materials showed no definite trend over a four week period. The deviation from anticipated results was considered to be a result of deficiencies in the technique, rather than interaction of the tracer with the bag.

4.6

Overall Accuracy and Functionality

Experiments were performed to determine the overall accuracy and functionality of the method. Tests involving the use of urine volumes of 100, 250, and 500 mls. urine in collection bags resulted in volumes by analysis of 101, 242, and 484 mls., or errors of 1, 3, and 3% respectively. Repeated use of the method and improved techniques show promise of reducing these errors to acceptable levels.

4.7

Chromium and Strontium as Alternative Tracers

Urine specimens were analyzed for chromium and for strontium, since these elements might also be feasible tracers, either as alternatives to the use of lithium, or as supplements to lithium in improving the accuracy of the method. The instrument response to chromium was 0.02 ppm., and to strontium 0.18 ppm.

5.0

RECOMMENDATIONS

The observations and results of the project have suggested certain areas that should be investigated in order to more fully understand the errors involved in the lithium tracer method, and to reduce error and increase reliability of the method. The following

5.0

(continued)

recommendations are made for consideration in the next phase of investigation to apply the tracer method for urine volume determinations:

5.1

Determine the Effect of Lithium Tracer Concentration on Urine Volume Error

Results of the accuracy tests in section 3.6 suggest a higher level of accuracy with 5 ppm. lithium concentration than at lower concentrations. Experiments should be performed which include a greater number of repetitions, and higher concentrations of lithium to determine effects on accuracy and repeatability, and to increase confidence in the results. Total instrument scatter at various concentrations should also be determined.

5.2

Minimum Mixing Time

Determine the minimum mixing time required for complete dispersion of the tracer in urine contained in the collection bag.

5.3

Minimum Degree of Dilution

Determine the minimum degree of dilution required to eliminate urine interference in the analysis and to prevent plugging of the sampling tube of the spectrophotometer.

5.4

Instrument Error

Investigate possibilities for reducing instrument error.

5.5

Chromium and Strontium as Tracers

Consider the use of chromium and strontium as tracers. In addition,

5.5

(continued)

consider the possible use of two or more elements, such as lithium and chromium to be included as tracers, with analysis of both elements. Determine effects of multi-tracers on accuracy.

5.6

Solvents as Diluents

Consider the use of solvents as diluents for urine, and determine effects on dilution ratio requirements to prevent plugging of the spectrophotometer sampling tube, as well as effects on accuracy and instrument scatter.

In response to request by R. L. Sauer, contract monitor, the following information is provided as supplement to Final Report of Contract NAS 9-10528.

1. Theoretical and Actual Tracer Concentrations in Dispersion Experiments

Theoretical determinations of equilibrium tracer concentrations involved in experiments 3.4.2 and 3.4.3 cannot be accurately established because the experimental procedures did not include accurate volumetric measurement of tracer and/or urine volumes. Since the primary purpose of the tests was to determine the rate of tracer dispersion, rather than actual end point analysis, volumetric measurements were considered to be irrelevant to the problem, and, therefore, were only approximate.

The experiment of 3.4.1, however, is somewhat more amenable to being analyzed because of the nature of the experimental procedure, since the tracer solution was pipetted onto the urine surface. The urine, however, was not accurately measured volumetrically, and, therefore, may be expected to account for a deviation between theoretical and actual values. The following is shown as a derivation of the predicted equilibrium tracer concentration involved in experiment 3.4.1:

5 ml. 1000 ppm Li in 400 ml. = 405 ml. total

5000 μ g/405 ml. = 12.33 ppm theo. conc.

Removed in Sampling: 30 ml. at ave. 2.35 ppm conc.

30 x 2.35 = 70.5 μ g Li removed

5000 μ g - 70.5 = 4930 μ g remaining

405 ml. - 30 = 375 ml. remaining

$\therefore \frac{4930}{375} = 13.1$ ppm theo. adjusted conc. Urine vol. approx.

(12.69 ppm actual)

CASE FILE
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2. Method of Mixing in 3.6.2.1 A.

The urine was mixed with the tracer by closing the zipper of the bag and shaking by hand at a rate of approximately 120 shakes per minute.

3. Method of Urine Addition in Bag Tests

(a) Bag Test of 3.6.1.1.A

A glass funnel was attached to the inside bore of the rubber stopper used to seal to the adhesive of the bag opening. The urine was added to the bag by pouring into the funnel from the volumetric flasks used for measuring urine volumes.

(b) Bag Tests of 3.6.2.1.A

The urine was poured directly into the bags from the volumetric flasks used to measure urine volumes.

(c) Bag Tests of 3.4.3.1.A

The urine was added to the bag by slowly pouring from a beaker. Pouring produced little agitation in the solution. However, it will be noted that during the pouring in this particular test, some of the solution overflowed from the bag opening due to difficulty experienced in holding the opening in an open condition in a bench-clamp arrangement. After pouring, and after a slight adjustment of the clamp, the bag was left in an undisturbed condition for the duration of the test.

4. Recommendations Related to Estimated Tracer Concentration for Maximum Sensitivity

The results of sections 3.6.1 and 3.6.2 would suggest that the analysis of the lithium tracer at approximately 5 ppm concentration

resulted in better accuracy than at lower concentrations. If these results can be considered real (that is, results are based on a limited number of tests), then it would appear that accuracy of the order of $\pm 1\%$ or greater should occur with tracer concentrations of 5 ppm and higher.

A 5 ppm concentration in 500 ml. void volume, solution diluted 10:1 for analysis, would require:

$$5 \mu\text{g/ml.} \times 500 \text{ ml.} \times 10 = 25 \text{ mg Li (153 mg LiCl)}$$

This amount of tracer would result in analytical concentrations of tracer in various void volumes as follows:

Urine Void Vol. ml.	Li Conc. in Urine ppm	Analytical Conc. (Diluted 10:1) ppm
100	250	25
250	100	10
500	50	5