OPEN

CR 115262

September 1, 1971

FINAL REPORT

TRACER TECHNIQUE FOR URINE VOLUME DETERMINATION AND URINE COLLECTION AND SAMPLING BACK-UP SYSTEM

CONTRACT NAS 9-11503

Submitted to: NASA Manned Spacecraft Center
Mr. R. L. Sauer, DC7
Preventive Medicine Division
Houston, Texas 77058

Submitted by: Life Support Systems Group
Whirlpool Corporation
300 Broad Street
St. Joseph, Michigan 49085

N72-12025 (NASA-CR-115262) TRACER TECHNIQUES FOR URINE VOLUME DETERMINATION AND URINE COLLECTION AND SAMPLING BACK-UP SYSTEM Final Report R.V. Ramirez (Whirlpool Corp.) 1 Sep. 1971 36 p CSCL 06P G3/04
Unclas 10111
FA (NASA CR OR TMX OR AD NUMBER) (CATEGORY)

Prepared by:

Ruben V. Ramirez, Chemist
Life Support Systems Group
Whirlpool Corporation

Approved by:

Norman G. Roth, Ph.D., Dir.
Life Support Systems Group
Whirlpool Corporation

6.3.2 (continued)

In close observation, minute particles of possible capsule material could be seen in the urine. Tests were prepared to check for possible interference of preservative in gelatin capsule with lithium tracer analysis.

Three containers with 5 ml of lithium stock solution were oven dried, and 600 ml (room temperature) urine was added to each. Sodium omadine (0.63 gm) in capsule form was deposited in each container and mixed two to three minutes. Urine samples were drawn and diluted for analysis. See Table 6.3.2.A.

6.3.3 Results

See Table 6.3.2.A on the following page for test results.

TABLE 6.3.2.A

ANALYSIS FOR INTERFERENCE OF CHEMICAL PRESERVATIVE WITH LITHIUM TRACER

Mixing Time (Minutes)	Urine Vol. (mls)	Li Conc. in Vessel (ppm)	Li Conc. Diluted Urine 1:50 (ppm)	Li Conc. by Analysis (ppm)	Error ML ±%	Vol. Deter. by Analysis (Mls)
2 to 3	600	30	1.00	0.99	-6 -1.0	594
2 to 3	600	30	1.00	0.99	-6 -1.0	594
2 to 3	600	30	1.00	0.99	-6 -1.0	594
2 to 3	600	30	1.00	0.99	-6 -1.0	594
2 to 3	600	30	1.00	0.99	-6 -1.0	594
2 to 3	600	30	1.00	0.99	-6 -1.0	594
2 to 3	600	30	1.00	1.00	0 0	600
2 to 3	600	30	1.00	0.99	-6 -1.0	594
2 to 3	600	30	1.00	1.00	0 0	600

6.3.4 Conclusion

The test results seen in Table 6.3.2.A indicate that the encapsulated chemical urine preservative does not interfere with lithium tracer analysis, and the level of error is comparable to results obtained in other tests.

URINE VOLUME DETERMINATION STUDY

1.0 INTRODUCTION

This study was initiated to further investigate the feasibility, functionality and overall accuracy of the use of lithium as a chemical tracer in urine, thereby providing a means for indirect determination of total urine volume by atomic absorption spectrophotometry method. Experiments were conducted to investigate the parameters of instrumentation, tracer concentration, mixing times, and methods for incorporating the tracer material in the urine collection bag, and to refine and optimize the urine tracer technique to comply with Skylab scheme and operational parameters of $\pm$ 2% of volume error and $\pm$ 1% accuracy of amount of tracer added to each container. In addition, a back-up method for urine collection and sampling system was developed and evaluated. This back-up method incorporates the tracer technique for volume determination in event of failure of the primary urine collection and preservation system. One chemical preservative was selected and evaluated as contingency chemical preservative for the storage of urine in event of failure of urine cooling system.

2.0 PHASE I

2.1 Purpose:

This phase evaluated techniques for the improvement of the accuracy of the tracer method. This phase also determined optimum tracer concentration and mixing time. Consideration was given to possible use of multi tracers, solvents as diluents for analysis and techniques for reducing instrument scatter and error.

2.2 Task a

2.2.1 Description of Task

Determine spectrophotometer scatter and error with concentration of lithium tracer in urine of 1, 2, 5, 10, 20, 30 and 50 ppm.

2.2.2 Method

Tracer analyses were conducted with Perkin Elmer Model 403 Atomic Absorption Spectrophotometer with air-acetylene fuel mixture system. The instrument was prepared and peaked for optimum element detection in the same manner each time to maintain best performance and reduce scatter and error. The detection limit of the spectrophotometer is 0.5 to 5 μg/ml. Instrument sensitivity is considered at 0.03 μg/ml lithium and linear up to concentrations of approximately 2 μg/ml in aqueous solution. Aqueous lithium tracer was delivered volumetrically in all cases and dilutions were prepared in volumetric glassware with deionized water. All samples withdrawn for analysis were done with volumetric pipettes. Although there are several variables that may introduce errors in analysis, most of the potential variables were checked in the laboratory against suitable standards. Tracer salt purity was checked against standard analytical solutions of lithium from Harleco Company used to calibrate the spectrophotometer.

Experiments were conducted with urine diluted (1:10) with water and urine containing tracer at various concentrations to investigate instrument scatter and error and to improve accuracy of lithium tracer determination by atomic absorption. The test samples were aspirated at several different rates to evaluate the stability of the nebulizer and efficiency of the sample tube to deliver a constant flow of solution to the burner and remain free from clogging. It was noted that

2.2.2 (continued)

tube clogging could be eliminated by using diluted urine. Instrument response was improved and scatter was reduced by maintaining a constant rate of aspiration. An aspiration rate of 6 to 8 ml per minute was found satisfactory and used for all subsequent analysis. Lithium concentrations of 1 to 2 ppm in urine diluted (1:10) showed the least instrument scatter, and percent error was reduced. Precision of instrument was also greatly improved by the use of dilute aqueous test sample.

2.2.3 Results

The amount of tracer is primarily determined by the accuracy and detection limits of the instrument. The highest level of accuracy was seen of lithium concentration in 1 to 2 ppm range prepared for atomic absorption analysis. When aspiration rate of diluted urine is maintained constant, instrument scatter and level of error was definitely improved.

2.2.4 Conclusion

The conclusion is that lithium concentration in diluted urine should be in 1 to 2 ppm range to maintain high level accuracy and precision.

The findings also suggest that the tracer concentration in the urine collection bag should be at least 30 mg lithium when maximum urine volume of 3,000 ml is considered, or an average of 10 mg lithium per liter of urine. Therefore, one ml available for analysis, diluted 1:10, will contain 1 ppm Li.

2.3 Task b

2.3.1 Description of Task

Determine effect of mixing time of 1, 2, 5, 10 and 30 minutes on analytic scatter and error, at lithium or other tracer concentrations of 1, 2, 5, 30 and 50 ppm. A standardized method of mixing shall be developed and

2.3.1 (continued)

utilized for these experiments. The effects of a urine temperature at 36° F in mixing will be determined.

2.3.2 Method

Lithium chloride stock solution (1,000 ppm Li) was prepared in the laboratory with deionized water. Lithium standard from Harleco was used to calibrate the spectrophotometer. The experiments were prepared by delivering 5 ml of 1,000 ppm Li and adding 495 ml of pooled chilled urine (36° to 40° F) in volumetric flasks. The chilled contents were mixed for 1, 2, 5, 10 and 30 minutes, withdrawing an aliquot of urine for analysis after each mixing time. Mixing was accomplished by inverting the vessel approximately 30 times per minute.

Ten mls of the urine-lithium contents were diluted with deionized water and 10 ml of potassium chloride solution (10,000 ppm KCL) in 100 ml volumetric flasks. This placed the lithium concentration of dilute urine (1:10) at 1 to 2 ppm.

The potassium chloride solution was used as a buffer agent to help reduce scatter and interference.

The tests were conducted by inserting the sample tube from the spectrophotometer directly into the contents of the flask and aspirated at a constant rate of approximately 6 to 8 ml per minute. The concentrations shown in all experiments in this report were the average of 100 samplings, obtainable with this model spectrophotometer. Standard solutions were prepared by diluting lithium nitrate (1,000 ppm Li) spectrographic standard with water and potassium chloride solution, the same as in all test samples. This offered a direct comparison of the test samples with a reliable standard.

2.3.3 Results

The results of this experiment (Table 2.3.3.A and Table 2.3.3.B) show that a complete dispersion of lithium tracer in chilled urine can be attained by proper mixing.

TABLE 2.3.3.A

Mixing Time-Mixing Method vs. Accuracy

Mixing Time	Li Std. 1 ppm conc.	Li 1 ppm Conc. Urine Diluted 1:10	Li Conc. By Analysis	Percent Error
1 Minute	1.00	1.00	1.00	0%
2 Minutes	1.00	1.00	1.01	1%
5 Minutes	1.00	1.00	1.02	2%
10 Minutes	1.00	1.00	1.02	2%
30 Minutes	1.00	1.00	1.01	1%

TABLE 2.3.3.B

Mixing Time-Mixing Method vs. Accuracy

Mixing Time	Li Std. 2 ppm conc.	Li 2 ppm Conc. Urine Diluted 1:10	Li Conc. By Analysis	Percent Error
1 Minute	2.00	2.00	2.01	0.5%
2 Minutes	2.00	2.00	2.04	2%
5 Minutes	2.00	2.00	2.03	1.5%
10 Minutes	2.00	2.00	2.02	1%
30 Minutes	2.00	2.00	2.01	0.5%

2.3.4 Conclusion

A homogeneous mixture is evident in the first or second minute of mixing. The findings suggest that at least one (1) full minute is necessary to attain optimum dispersion of lithium tracer in pooled chilled urine.

2.3.5 Prototype Urine Collection Bags

Experiments were prepared as in 2.3.2 except that 5 mls of lithium (1,000 ppm Li) were delivered into the prototype bag through the orifice provided. Four-hundred ninety-five (495) mls of pooled chilled urine were delivered volumetrically, and the contents were mixed for one (1) full minute. The mixing was done manually by closing the orifice and holding the bag horizontally between the hands. With a slight lateral movement of the hands and arms the contents were sloshed from side to side in the bag. Although some foaming of the chilled urine was evident, there was no indication of interference with dispersion of tracer nor at time of sample extraction for analysis.

Mechanical methods for mixing are available; however, mixing manually proved satisfactory. Urine test samples were withdrawn soon after the mixing stopped and diluted as in 2.3.2.

2.3.6 Results

The results of this experiment (Table 2.3.6A) show that a good dispersion of lithium in urine in prototype urine collection bags can be attained by manually mixing the contents for one (1) full minute.

See Table 2.3.6A on next page.

2.3.6 (continued)

TABLE 2.3.6A

Mixing Time-Mixing Method vs. Accuracy

Mixing Time (Minutes)	Urine Volume	Li Std. 1 ppm Conc.	Li Conc Dilute Urine (1:10) ppm	Li Conc. By Analysis ppm	Percent Error
1	495	1.00	1.00	1.00	0%
1	495	1.00	1.00	1.00	0%
1	495	1.00	1.00	0.99	-1.0%
1	495	1.00	1.00	1.00	0%
1	495	1.00	1.00	0.99	-1.0%
1	495	1.00	1.00	1.00	0%
1	495	1.00	1.00	0.99	-1.0%
1	495	1.00	1.00	1.00	0%
1	495	1.00	1.00	0.99	-1.0%
1	495	1.00	1.00	1.00	0
1	495	1.00	1.00	0.99	-1.0%
1	495	1.00	1.00	1.00	0%

2.3.7 Conclusion

In order to obtain a satisfactory dispersion of lithium in pooled chilled urine, the contents should be mixed for one (1) full minute. The mixing can be readily accomplished by the manual method stated in 2.3.5. The results in Table 2.3.6.A are consistent and within the allowable volume error.

2.4 Task c

2.4.1 Description of Task

Determine the effects of the use of acetone, methanol, and ethanol as diluents for the analysis of tracer in urine, using the tracer concentration and mixing time which shows the minimum scatter and error as determined in Task a and Task b.

2.4.2 Method

Tests were conducted to establish the use of organic solvents as diluents for tracer analysis and also reduce instrument background interference.

Aqueous test solutions were prepared from each of the solvents in 2.4.1 and aspirated at constant rate.

In every case the dilute solvents caused considerable flame effects and interference. The level of scatter and error that was experienced was higher than the limits allowable. The use of solvents may also constitute a potential safety hazard when mixed with the burner gases and ignited.

2.4.3 Results

The use of organic solvents as diluent for lithium analysis contributed a high degree of interference and instrument scatter and was definitely no improvement over deionized water as the diluent.

2.4.4 Conclusion

Organic solvents as diluents for lithium tracer were considered unacceptable because of the high interference contributed.

The use of deionized water is by far superior to solvents.

2.5 Task d

2.5.1 Description of Task

Determine the effects of the use of chromium and lithium-chromium tracer on analytical scatter and error, using tracer concentration and mixing time as determined in Task a and Task b.

2.5.2 Method

Tests of a qualitative nature were conducted to determine the possible use of chromium and lithium-chromium tracer for urine volume determination. Since the level of error found in chromium urine tests was higher than that allowed, the tests with chromium-lithium showed considerable increase in error as a multi-tracer method. It was also learned that a complete change-over of instrument adjustments and mechanics to analyze both tracers was necessary in the multi-tracer scheme, therefore creating a greater instrument error.

2.5.3 Results

The results of these tests indicated that the use of chromium tracer, alone or in combination, would incur a percent error greater than $\pm$ 2%.

2.5.4 Conclusion

The use of this tracer scheme is not acceptable because of error magnification.

2.6 Task e

2.6.1 Description of Task

Investigate possibilities for reducing scatter and error caused by the analytical procedure and instrumentation. Examine optical filters, instrument pick-up tube depth in solution, pick-up tube length, and flame effects.

2.6.2 Method

2.6.2.1 Optical Filters

Optical filters for the spectrophotometer were examined to determine if scatter and error could be reduced through the use of a single or multi-filter system. The examination ruled out the external mounting of filter to improve instrument procedure. However, it was learned that the use of internal filter system incorporated in this model considerably improved instrument performance.

2.6.2.2 Burner

The proper alignment of the four-inch single slot burner also increased the performance. This consisted of horizontal positioning of the burner in line with the light path in the combustion chamber of the spectrophotometer.

2.6.2.3 Flame effect

Best performance was also experienced with an even oxidizing flame of average height.

2.6.2.4 Potassium Chloride Solution

A solution of potassium chloride (10,000 ppm KCL) was prepared by dissolving 18.5 gm potassium chloride crystals in a liter of water. The ten ml. volume used in diluting the urine (1:10) in a 100 ml flask acted as a buffer agent and generally improved the analytical procedure. This solution was used in preparing the test and reference standard solutions for analysis.

2.6.2.5 Instrument Pick-Up Tube

Tests were made with the instrument sampling tube at different depths in the test solution. Depths of 1/4-inch, 1/2-inch, 1-inch and 2-inches from the surface were investigated. The depth of sampling tube in solution showed no significant evidence of influencing the analysis. However, it should be pointed out that the length of sampling tube does change the rate of aspiration, and it is suggested that operating aspiration rates be established with each tube size used. A 7-inch long polyethylene tubing that would fit securely over the nebulizer stem was used successfully for these tests. An aspiration rate of 6 to 8 mls per minute proved satisfactory with this tube, and has since been used in analytical procedures.

2.6.2.6 Volumetric glassware

In all procedures applicable to the tracer technique, volumetric glassware was employed. The glassware was nitric acid washed and properly rinsed before use. Precision, quality Class A, serialized, volumetric pipettes purchased from Fisher Scientific Company were used to maintain the required level of accuracy, $\pm$ 1% of amount of tracer added to each container. Proper analytical and quantitative procedures were employed at all times.

2.6.2.7 Lithium Stock Solution (6,000 ppm Li)

The lithium stock solution was prepared in the laboratory from Lithium Chloride, powder, Fisher Scientific certified. The powder was oven dried overnight, and 36.5856 gms of LiCl were carefully weighed and diluted in a liter of deionized water. Therefore, one ml stock solution contained 6,000 ppm or 6 mgs Li. It should be pointed out that a standard prepared from the same stock solution used for the urine collection bags will satisfactorily fill the standard role and further reduces tracer error.

2.6.2.8 Spectrographic Lithium Standard

The experiments conducted in the entire study for this contract were directly compared against a reliable spectrographic standard of Lithium Nitrate (1,000 ppm), diluted to the desired concentrations with water and potassium chloride, same as in tests in 2.3.2. The standard is a product of Harleco Company.

2.6.2.9 Urine Standard

A urine standard was not considered for these experiments because of potential variables that may exist. However, a synthetic urine standard could probably be used as reference standard, but no synthetic urine standard was used in this study.

2.6.2.10 Results

The findings of Task e suggest that employing proper analytical procedure and good control over the mechanics of instrumentation, scatter and error are reduced.

2.6.2.11 Conclusion

Scatter and error are reduced by using proper procedure and control and by preparing standard to calibrate instrument from same lithium stock solution used in collection containers.

2.7 Task f

2.7.1 Description of Task

Conduct these tests in such a manner that mass balance computation can be made in order to determine total recoverability of tracer. This technique will enable a continuing evaluation to be made as to possible loss of tracer through sorption processes.

2.7.2 Method

2.7.2.1 Sorption Study using UCMSS Prototype Bag and Filter Material

The prototype Teflon bags were manufactured by Dilectrix Corporation, and a representative sample of the filter material used in bag scheme was supplied separately.

The tests were prepared by delivering 5 ml of stock lithium solution (2.6.2.7) to each bag volumetrically through the bag orifice. Three-inch squares of the filter material were placed in glass containers, and 5-ml of stock solution were delivered to each vessel. The bags and the glass containers were oven dried overnight at approximately 110° F with circulating air stream. Six-hundred (600) mls of pooled chilled urine (36° to 40° F) were added to each container and mixed for one minute.

Samples were extracted for analysis and diluted (1:50). The Sorption Study was prepared starting with Week 0 through Week 5. During the interim between weeks, the test bags and container were kept refrigerated at approximately 36° F. At the end of each week the contents were mixed again for one minute, and samples were extracted for analysis.

TABLE 2.7.2.1.A

UCMSS PROTOTYPE BAG SORPTION STUDY

Week	Bag 1			Bag 2			Bag 3		
	A	B	C	A	B	C	A	B	C
0	1.00	0.99	1.00	0.99	0.99	0.99	0.99	0.99	0.99
1	1.00	1.00	1.00	1.01	1.01	1.01	1.00	1.00	1.00
2	0.99	1.00	0.99	1.00	1.00	1.01	1.00	1.00	0.99
3	0.99	0.99	0.99	0.99	1.00	1.00	1.00	1.00	1.00
4	1.00	1.00	1.01	0.99	0.99	0.99	1.01	1.01	1.01
5	1.01	1.01	1.01	1.00	1.00	1.00	1.01	1.00	1.00

TABLE 2.7.2.1.B

UCMSS PROTOTYPE HYDROPHOBIC FILTER MATERIAL SORPTION STUDY

Week	Container 1			Container 2			Container 3		
	A	B	C	A	B	C	A	B	C
0	0.99	0.99	0.99	1.01	0.99	0.99	1.00	0.99	1.00
1	1.01	1.00	1.00	1.01	1.01	1.00	1.00	0.99	1.00
2	1.00	1.01	1.00	1.01	1.01	1.00	1.00	0.99	1.00
3	1.01	1.02	1.02	1.00	1.00	1.00	1.00	0.99	0.99
4	1.00	0.99	1.00	1.01	1.00	1.00	1.00	1.00	1.00
5	1.00	1.00	1.01	1.01	1.02	1.01	1.00	1.00	0.99

Conditions:
Lithium conc. in each container - 30 mgs
Diluted urine for analysis - 1:50
Lithium standard - 1 ppm
Readings are in ppm

2.7.3 Results

Table 2.7.2.1.A and 2.7.2.1.B show the results of the analyses conducted to evaluate the possible loss of tracer through sorption process of bag materials. The lithium concentration for all the tests of this task should have read 1.00 ppm. The error can be assumed to be that of instrument and laboratory technique rather than loss of tracer through sorption processes. The prototype UCMSS Bag material did not appear to produce any appreciable influence upon or alternation of lithium concentration.

2.7.4 Conclusion

The conclusion of the experiment was that no appreciable reaction occurred between bag material and tracer concentration that would contribute loss of tracer.

3.0 PHASE II

3.1 Purpose

This phase evaluated methods for addition of tracer to urine collection bags in order to recommend a method for the mockup Skylab UCMSS bag and the contingency bag. Consideration was given to various techniques for incorporating precise quantities of tracer in the bags.

3.2 Tasks a and b

3.2.1 Description of Task

Methods of adding tracer to the mockup Skylab UCMSS bag were studied and evaluated to attain the required accuracy of the amount of tracer added to each container.

3.2.2 Method

3.2.2.1 Tablets, impregnated wafers, or capsules concepts were evaluated as methods of adding tracer to the urine containers. A laboratory tablet press was used in attempt to produce a tablet that would contain 30 mgs Lithium $\pm$ 1%. It was found that lithium chloride powder attracts and absorbs moisture from the air at a rapid rate and that it would be impossible to manage the compound as a tablet or powder without introducing a sizable error because of the salt's deliquescent characteristic.

Outside sources that were contacted felt that the $\pm$ 1% tolerance would be impossible to meet as a tablet or capsule and that a more realistic tolerance would be approximately $\pm$ 5%.

3.2.2.2 Aqueous Lithium Tracer

The deliquescent characteristic of lithium chloride led to preparation and management of Li tracer from a stock solution to help maintain

3.2.2.2 (continued)

± 1% tolerance of tracer content in the urine containers.

Two separate types of tests were conducted to evaluate the method and accuracy of introducing the tracer in mockup prototype UCMSS bags and contingency bags. Precision quality volumetric pipettes that meet NBS tolerance requirements were used to deliver the tracer.

The tracer was delivered by inserting pipette in the orifice provided in mockup prototype bag and through the flapper valve of the 24-hour contingency bag.

Test I - The bags contained predelivered tracer in aqueous state.

Test II -The bags contained predelivered tracer solution and were oven dried at approximately 110° F in an air stream to evaporate the water.

Tracer addition was made with a 5 ml volumetric pipette with accuracy of ± 0.3%.

See Table 3.2.2.2.A on the following page.

3.2.2.2 (continued)

TABLE 3.2.2.2.A

Test I: ACCURACY OF TRACER ADDITION TO URINE CONTINGENCY BAG

Mixing Time (Minutes)	Total Urine Vol. (Mls)	Li Conc. in bags (ppm)	Li Conc. Diluted Urine (ppm)	Li Conc. by Analysis (ppm)	Percent Error
1	495	5	1.00	0.99	1.0 %
1	495	5	1.00	1.00	0
1	495	5	1.00	0.99	1.0 %
1	595	30	1.00	1.00	0
1	595	30	1.00	1.01	1.0 %
1	595	30	1.00	1.00	0
1	595	30	1.00	1.02	2.0 %
1	595	30	1.00	1.00	0
1	595	30	1.00	1.00	0

Test II: ACCURACY OF TRACER ADDITION TO URINE UCMSS PROTOTYPE BAG

1	600	30	1.00	1.00	0
1	600	30	1.00	0.99	1.0 %
1	600	30	1.00	1.00	0
1	600	30	1.00	1.00	0
1	600	30	1.00	1.00	0
1	600	30	1.00	1.00	0
1	600	30	1.00	1.00	0
1	600	30	1.00	1.02	2.0 %
1	600	30	1.00	0.99	1.0 %

3.2.2.3 Results

The test results in Table 3.2.2.2.A indicate that adding tracer by method suggested in 3.2.2.2 is highly reliable and reproducible.

Tracer in the liquid or liquid-dehydrated form can be placed into the urine collection containers with the consistency and accuracy required. It should be pointed out that tracer could be added at the time of container manufacturing or after completion of container as long as an opening is available to allow tracer addition.

3.2.2.4 Water Soluble Film

It was learned that dehydrated lithium chloride tracer in sealed urine collection bags was collecting moisture and forming a liquid. There was concern that the liquified tracer would be lost in handling and storage of urine bags. It was decided that Methyl Cellulose, a water soluble film with trade name of Methocel or Edisol-M, could be used to bind the tracer and hold it from flowing. This film is clear, colorless, odorless, and the components of the film have FDA approval for food use. The syrupy consistency formed by the tracer and Methocel showed a cohesive characteristic that would help keep the tracer in place inside the bag.

It was decided that the maximum quantity of Methocel film to be used was 0.5 gm per urine bag.

Tests were prepared by placing 0.5 gm of Methocel film and 5 ml of lithium stock solution (30 mgs Li) in containers and oven drying at 110° F. The tracer was bound by the Methocel by this method and remained fixed, even though the container was left open to room air with considerable humidity for several days. Pooled chilled urine

3.2.2.4 (continued)

(600 mls) was added to each container and stored in refrigerator (36° F) for approximately four (4) hours with no mixing. At end of storage period the contents were examined. The Methocel-tracer had dissolved completely after this time. The containers were mixed for one (1) minute by swirling contents in vessel.

3.2.2.5 Results

The results of this test, seen in Table 3.2.2.5.A, indicate that the lithium bound by methocel was completely released and did not interfere with analysis.

TABLE 3.2.2.5.A

LITHIUM TRACER-METHOCEL ANALYSIS

Mixing Time (Minutes)	Total Urine Vol. (Mls)	Li Conc. in Vessel (ppm)	Li Conc. Diluted Urine 1:50 (ppm)	Li Conc. by Analysis (ppm)	Percent Error
1	600	30	1.00	1.00	0
1	600	30	1.00	1.02	2.0
1	600	30	1.00	0.99	1.0
1	600	30	1.00	1.00	0
1	600	30	1.00	1.00	0
1	600	30	1.00	1.00	0
1	600	30	1.00	1.01	1.0
1	600	30	1.00	1.00	0
1	600	30	1.00	1.01	1.0
1	600	30	1.00	1.00	0
1	600	30	1.00	1.02	2.0
1	600	30	1.00	1.01	1.0

3.2.2.6 Conclusion

The best method found for adding tracer to urine collection container is by volumetric delivery of a known amount of aqueous lithium tracer stock in order to maintain the level of accuracy and reliability desired. The use of Methocel water soluble film is considered a feasible and reliable method to bind and keep tracer in place, and yet completely release the tracer for analysis.

4.0 PHASE III

4.1 Purpose

The purpose of this phase was to investigate, evaluate, and develop urine collection and sample methods as contingency backup systems for the current Skylab System.

4.2 Task a

4.2.1 Description of Task

Develop a 24-hour urine collection container as backup for individual urine containers, to be compatible with onboard urine chilling aparatus in the Skylab UCMSS and with the overboard urine dump capability.

4.2.2 Method

A container, fabricated with polyurethane sheet film and having a rigid urine receiver, and roll-on cuff crewman interface, is employed to collect the total voided urine of an individual for a 24-hour period. After this period, the complete unit is discarded and replaced with a fresh assembly. Figure 4.2.2.(a) is a sketch of the contingency urine collection and sampling system.

4.2.3 Results

The roll-on cuff and receiver check valve is employed in the present Apollo Manned Spacecraft Urine Collection System. Therefore, the receiver arrangement has been proven to work at Zero Gravity. The container has a flat envelope of 13-inch square. It is 3/4-inch thick, containing 2,000 ml of liquid, and 1-5/8-inch thick, containing 3,000 ml of liquid.

CONTINGENCY URINE COLLECTION AND SAMPLING SYSTEM

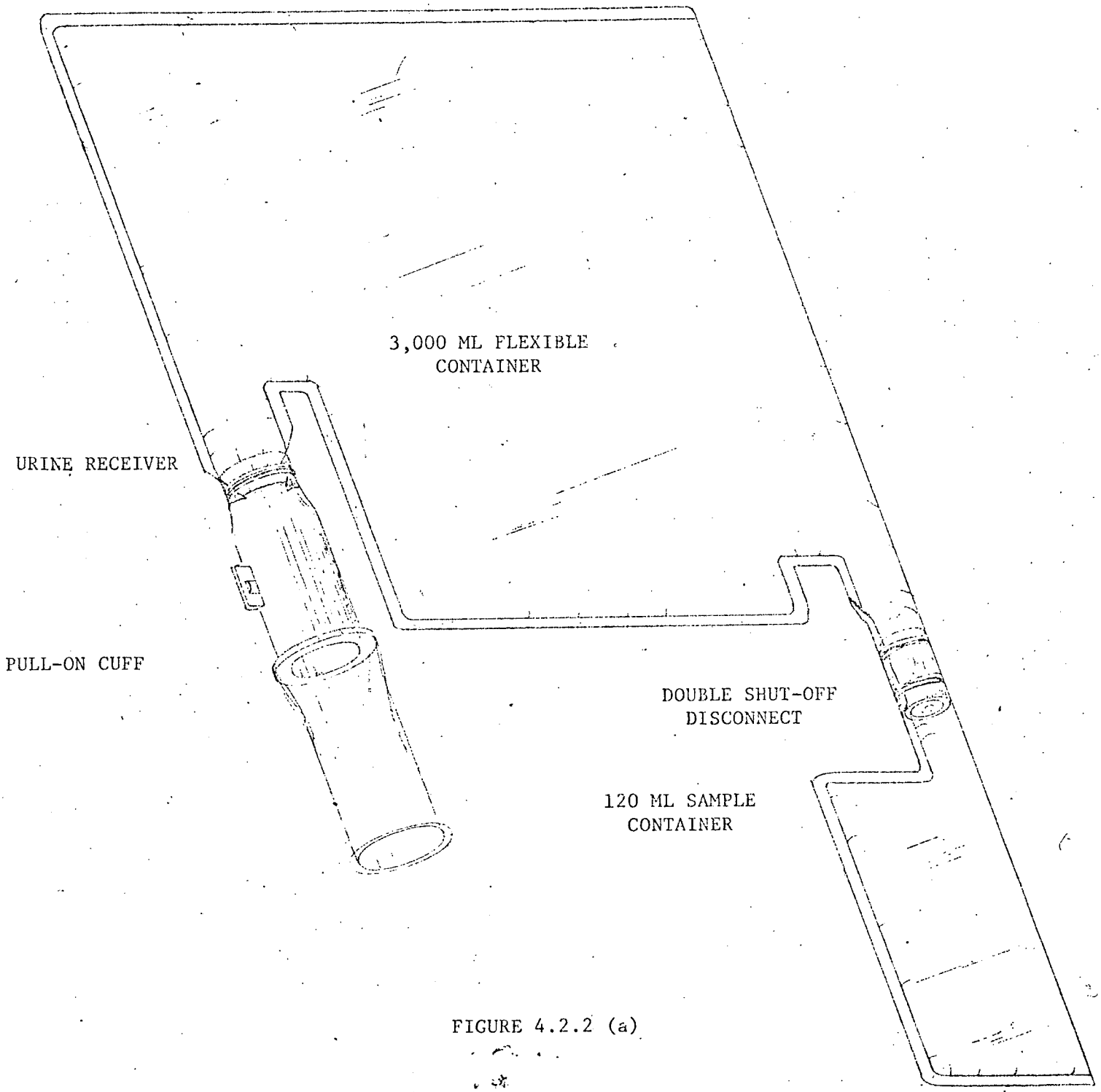


FIGURE 4.2.2 (a)

4.2.4 Conclusion

This design is compatible with all other requirements of this contract, will collect urine as voided, and will contain an average one man-day urine excretion with considerable margin within an envelope compatible with the chilling aparatus and other interfaces.

4.3 Task b

4.3.1 Description of Task

Develop a urine sample collection method integral with the container of Task A. The system shall be compatible with onboard preservation system (freezing contained sample in a compartment).

4.3.2 Method

A small container, fabricated with polyurethane sheet film, is attached to the 24-hour collection container through a double shut-off disconnect.

The disconnect is two small poppet valves placed end to end in a rubber tubing. The valves and passage between the two containers are open when the valve ends are squeezed together. At the end of each collection period, the valves shall be opened and 120 ml of collected urine shall be transferred to the sample container. After transferring a homogeneous sample, its container is disconnected from the collection container by cutting through the rubber tubing between the two poppet valves with a scissors or knife.

Individual sample containers fabricated with a reliable technique would not stow in individual compartments 2 x 2 x 2-3/8. However, three tubular containers, each filled with 120 ml, will stow in a

4.3.2 (continued)

2 x 2-3/8 x 6+" compartment formed by removing two partitions between three individual 2 x 2 x 2-3/8" compartments.

Although the sample container has capacity greater than 120 ml, one sample container restraint per mission shall be provided to limit capacity to 120 ml during transfer. The two poppet valves discussed in this section can be retained in a squeezed position by this restraint during transfer of sample quantity.

4.3.3 Results

It was verified that three sample containers containing 120 ml each of urine can be stowed within an envelope of 2 x 2-3/8 x 6. Three sample containers, complete with valves, and a scale stowage compartment were fabricated for verification testing.

The function of the disconnect was verified. Components from the Apollo In-Suit Drinking Device "Tilt-Valve" are utilized in this valve. Because flow through the valves is rather slow, it became inconvenient to physically maintain the squeezing force on valves during transfer. A mechanical device is provided in the restraint for this purpose.

4.3.4 Conclusion

A sample container integral with the contingency collection container Task A is feasible and will provide the function within the requirements and interfaces of the statement of work.

5.0 PHASE IV

5.1 Purpose

Utilizing the collection bags developed in Phase III and prototype UCMSS bags, experiments were performed to determine overall accuracy and functionality of best method and techniques developed in Phase I and II.

5.2 Task a.

5.2.1 Description of Task

Accuracy of the method was determined in the mockup UCMSS bags by placing 600, 2,000, and 3,000 ml of urine in the bags and determining the volume by analysis of the tracer.

5.2.2 Method

Five (5) mls of lithium stock solution were placed in each bag and oven dried. Urine was introduced and mixed for one full minute. Aliquots of urine were drawn and diluted for analysis. Lithium standard of 1 ppm and 1.5 ppm were used for the tests.

5.2.3 Results

The test results are seen in Table 5.2.3.A and indicate accuracy of tracer technique to be within the limits allowed.

TABLE 5.2.3.A

MOCKUP UCMSS BAGS - URINE VOLUME DETERMINATION

Mixing Time (Minutes)	Total Urine Vol. (mls)	Li Conc. in Bags (ppm)	Li Conc. Diluted Urine 1:50 (ppm)	Li Conc. by Analysis (ppm)	Error ML	Error ±%	Vol. Deter. by Analysis (Mls)
1	600	30	1.00	1.00	0	0	600
1	600	30	1.00	1.00	0	0	600
1	600	30	1.00	1.00	0	0	600
1	600	30	1.00	1.00	0	0	600
1	600	30	1.00	1.00	0	0	600
1	600	30	1.00	1.01	+6	+1.0	606
1	600	30	1.00	1.00	0	0	600
1	600	30	1.00	1.00	0	0	600
1	600	30	1.00	0.99	-6	-1.0	594
1	600	30	1.00	1.00	0	0	600
1	600	30	1.00	1.00	0	0	600
1	600	30	1.00	1.00	0	0	600
1	600	30	1.00	1.00	0	0	600
1	600	30	1.00	1.00	0	0	600
1	600	30	1.00	1.00	0	0	600
			Diluted Urine 1:10				
1	2000	30	1.5	1.51	+20	+1.0	2020
1	2000	30	1.5	1.52	+40	+2.0	2040
1	2000	30	1.5	1.51	+20	+1.0	2020
1	2000	30	1.5	1.51	+20	+1.0	2020
1	2000	30	1.5	1.51	+20	+1.0	2020
1	2000	30	1.5	1.51	+20	+1.0	2020
1	2000	30	1.5	1.49	-20	-1.0	1980
1	2000	30	1.5	1.49	-20	-1.0	1980
1	2000	30	1.5	1.49	-20	-1.0	1980
1	2000	30	1.5	1.49	-20	-1.0	1980
1	2000	30	1.5	1.48	-40	-2.0	1960
1	2000	30	1.5	1.49	-20	-1.0	1980
1	2000	30	1.5	1.50	0	0	2000
1	2000	30	1.5	1.50	0	0	2000
1	2000	30	1.5	1.50	0	0	2000
			Diluted Urine 1:10				
1	3000	30	1.0	1.01	+30	+1	3030
1	3000	30	1.0	1.02	+60	+2	3060
1	3000	30	1.0	1.02	+60	+2	3060
1	3000	30	1.0	1.02	+60	+2	3060
1	3000	30	1.0	1.01	+30	+1	3030
1	3000	30	1.0	1.02	+60	+2	3060
1	3000	30	1.0	1.01	+30	+1	3030
1	3000	30	1.0	1.01	+30	+1	3030

. . . CONTINUED ON NEXT PAGE

TABLE 5.2.3.A - continued

Mixing Time (Minutes)	Total Urine Vol. (mls)	Li Conc. in Bags (ppm)	Li Conc. Diluted Urine 1:50 (ppm) Urine Diluted 1:10	Li Conc. by Analysis (ppm)	Error ML	Error +%	Vol. Deter. by Analysis (Mls)
1	3000	30	1.0	1.01	+30	+1	3030
1	3000	30	1.0	1.01	+30	+1	3030
1	3000	30	1.0	1.02	+60	+2	3060
1	3000	30	1.0	1.01	+30	+1	3030
1	3000	30	1.0	1.00	0	0	3000
1	3000	30	1.0	1.00	0	0	3000
1	3000	30	1.0	1.01	+30	+1	3030

5.2.4 Conclusion

Urine volume determination by analysis of aqueous lithium tracer, dehydrated, can be considered as reliable and best method of maintaining accuracy tolerance required.

5.3 Task b

5.3.1 Description of task

Accuracy of method was determined in the contingency bags by placing 600, 2,000, and 3,000 ml of urine in the bags and determining the volume by analysis of the tracer.

5.3.2 Method

The same method used in 5.2.2 was applied for these tests. The tracer was introduced through the flapper valve of contingency bag.

5.3.3 Results

The results of these tests are listed in Table 5.3.3.A. The results are consistent and reproducible and the level of accuracy is within tolerance.

TABLE 5.3.3.A

CONTINGENCY BAGS - URINE VOLUME DETERMINATION

Mixing Time (Minutes)	Total Urine Vol. (mls)	Li Conc. in Bags (ppm)	Li Conc. Diluted Urine 1:50 (ppm)	Li Conc. by Analysis (ppm)	Error ML	Error ±%	Vol. Deter. by Analysis (Mls)
1	600	30	1.00	1.00	0	0	600
1	600	30	1.00	1.00	0	0	600
1	600	30	1.00	1.00	0	0	600
1	600	30	1.00	1.01	+6	+1.0	606
1	600	30	1.00	1.01	+6	+1.0	606
1	600	30	1.00	1.00	0	0	600
1	600	30	1.00	1.00	0	0	600
1	600	30	1.00	1.00	0	0	600
1	600	30	1.00	1.00	0	0	600
1	600	30	1.00	0.99	-6	-1.0	594
1	600	30	1.00	0.99	-6	-1.0	594
1	600	30	1.00	1.00	0	0	600
1	600	30	1.00	1.00	0	0	600
1	600	30	1.00	0.99	-6	-1.0	594
			Diluted Urine (1:10)				
1	2000	30	1.5	1.51	+20	+1.0	2020
1	2000	30	1.5	1.50	0	0	2000
1	2000	30	1.5	1.50	0	0	2000
1	2000	30	1.5	1.51	+20	+1.0	2020
1	2000	30	1.5	1.52	+40	+2.0	2040
1	2000	30	1.5	1.51	+20	+1.0	2020
1	2000	30	1.5	1.51	+20	+1.0	2020
1	2000	30	1.5	1.51	+20	+1.0	2020
1	2000	30	1.5	1.50	0	0	2000
1	2000	30	1.5	1.52	+40	+2.0	2040
1	2000	30	1.5	1.51	+20	+1.0	2020
1	2000	30	1.5	1.52	+40	+2.0	2040
1	2000	30	1.5	1.51	+20	+1.0	2020
1	2000	30	1.5	1.52	+40	+2.0	2040
1	2000	30	1.5	1.51	+20	+1.0	2020
			Diluted Urine (1:10)				
1	3000	30	1.0	1.01	+30	+1.0	3030
1	3000	30	1.0	1.01	+30	+1.0	3030
1	3000	30	1.0	1.01	+30	+1.0	3030
1	3000	30	1.0	1.01	+30	+1.0	3030
1	3000	30	1.0	1.02	+60	+2.0	3060
1	3000	30	1.0	1.02	+60	+2.0	3060
1	3000	30	1.0	1.01	+30	+1.0	3030

. . .Continued on next page

TABLE 5.3.3.A

CONTINGENCY BAGS - URINE VOLUME DETERMINATION

Mixing Time (Minutes)	Total Urine Vol. (mls)	Li Conc. in Bags (ppm)	Li Conc. Diluted Urine 1:10 (ppm)	Li Conc. by Analysis (ppm)	Error ML	Error ±%	Vol. Deter. by Analysis (Mls)
1	3000	30	1.0	1.02	+60	+2.0	3060
1	3000	30	1.0	1.01	+30	+1.0	3030
1	3000	30	1.0	1.00	0	0	3000
1	3000	30	1.0	1.01	+30	+1.0	3030
1	3000	30	1.0	1.00	0	0	3000
1	3000	30	1.0	1.02	+60	+2.0	3060
1	3000	30	1.0	1.01	+30	+1.0	3030
1	3000	30	1.0	1.01	+30	+1.0	3030

5.3.4 Conclusion

The tracer technique shows similar consistency, reproducibility, and accuracy in both the UCMSS prototype and contingency bags. The findings of these studies definitely suggest that urine volume determination by lithium tracer technique to be reliable, and that employing proper control and analytical procedure, the accuracy tolerance can be maintained.

6.0 PHASE V

6.1 Purpose

Possible chemical preservative for the storage of urine as contingency in event of failure of urine cooling system was selected, and effectiveness of urine preservation was determined.

6.2 Task a

6.2.1 Description of Task

Effectiveness of urine preservation was determined by aerobic plate counts of treated urine stored at 30° C. Urine treated with the chemical preservative was tested for interference with analysis of the tracer.

6.2.2 Method

Microbiological tests were prepared to determine the effectiveness of sodium omadine chemical preservative in pooled raw urine. The determination was based on the tube dilution assay in Difco Plate Count Agar. Pure cultures of Staphylococcus Aureus and Escherichia Coli were prepared to inoculate the urine and establish population base-line and kill off. Culture tubes were incubated at 30° C. Aerobic plate counts were made at Day 0, 2, and 7 of storage.

6.2.3 Results

Urine samples were treated with chemical preservative concentration ranging from 0.01 to 0.09%. The results showed that the effective preservative concentration range was between 0.06 to 0.09%. Based on these findings, the amount of preservative that is necessary to stabilize the urine is 0.06% by weight sodium omadine. Therefore, $0.60\ gm\ ^{+.03}_{-.00}$ sodium omadine would be necessary to stabilize 1 liter of pooled urine or 0.30 per 500 ml.

6.2.4 Conclusion

The conclusion is that sodium omadine can be considered an effective urine preservative and shows no apparent interference with lithium analysis.

6.3 Task b

6.3.1 Description of Task

Methods for adding preservative to urine collection containers were evaluated and developed.

6.3.2 Method

Several methods of introducing the urine preservative to urine collection containers were given consideration, such as:

(1) Deposit of chemical preservative in powder form, powder in pouch, or in tablet prior to final completion of bag;

(2) Predeposit aqueous preservative and dehydrate same as tracer;

(3) Pack the powder in gelatin capsules and deposit as needed after completion of urine bag through available port of entry.

The best and most practical method considered is to add the urine preservative packed in water soluble gelatin capsules made by Eli Lilly and Company. The capsule can be added to contingency bag via flapper valve port. The capsule is pushed through with minimum of effort.

Tests were conducted to determine solubility rate of preservative packed in gelatin capsule in fresh urine at room temperature. The results showed that the capsule would tend to cling to side of container. However, it was noted that within two minutes the capsule started to dissolve, and within five minutes lapse time, three-fourths of the capsule had dissolved, and the released preservative had dissolved. In less than one hour the entire contents had dissolved, without mixing, at room temperature.