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# EVALUATION OF TEST STRIPS FOR DETERMINING INORGANIC CONTAMINANTS IN FIELD WATER


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A prototype WQAS-PM (Water Quality Analysis Set-Preventive Medicine) has been assembled using off-the-shelf test strip technology for the analysis of arsenic, magnesium, chloride, sulfate and cyanide in the determination of the potability of Army field water. This prototype set has been evaluated by in-house operators and by military field units as to accuracy, time required to complete the analysis, ease of operation, and clarity of individual instructions. High temperature storage of these test kits has also been evaluated. The results of these evaluations as well as recommended modifications are presented.

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## INTRODUCTION

Waterborne diseases and contaminants pose threats to the health of U.S. Army soldiers and therefore become a significant operational concern to military commanders during field operatons. A family of simple, reliable, and rapid equipment to monitor health related water quality parameters has been proposed to provide for a determination of the potability of field water.[1] Included in this family of equipment is a Water Quality Analysis Set - Preventive Medicine (WQAS-PM) which will measure those inorganic constituents that could cause adverse health effects to soldiers in the field.[2]

Current field chemical water quality analysis capabilities used by the military are limited and outdated because they require wet chemistry procedures using chemicals with a limited shelf life. Therefore, an updated WQAS-PM to be used by preventive medicine personnel needs to be developed to measure those chemical parameters in field drinking water that can cause adverse health effects and performance degradation of military field personnel. Among these parameters are arsenic, magnesium, chloride, sulfate, and cyanide. The WQAS-PM will be useful for determining each of these parameters with detection limits and quantification well below the levels that can cause performance decrements and adverse health effects. Test strip technology currently exists for these parameters and appears to be the most promising from the standpoint of cost, reliability, ease of operation in the field, size, and weight considerations. A comparison of the detection ranges desired by the Army and ranges obtainable through current kit technology is given in Table 1.

Table 1. Detection ranges: proposed and currently available.

| Parameter | Proposed Detection Ranges [3] (mg/L) | Kit Technology [a] (mg/L) |
|---|---|---|
| arsenic | 0.02 - 10 | 0.1 - 3.0 |
| magnesium | 30 - 100 | 10 - 120 |
| chloride | 600 - 3000 | 500 - 3000 |
| sulfate | 100 - 3000 | 400 - 1600 |
| cyanide | 2 - 20 | 1 - 30 |

[a] See Appendix I

A prototype for this water quality analysis set consisting of off-the-shelf test kits has been evaluated both by in-house operators and by military field units according to accuracy, time required to complete the analysis, ease of operation, clarity of individual instructions, and capabilities for storage under high temperature extremes. This prototype along with minor modifications will be sent to field units in an effort to provide an interim fix for field detection deficiencies. Meanwhile, developmental efforts will continue to improve test strip detection and shelf life.

## EXPERIMENTAL

**Materials.** Test strips and procedures (see Appendix I) evaluated included Chloride (Merckoquant 10079), Cyanide (Merckoquant 10044), Sulfate (Merckoquant 10019),

Arsenic (Merckoquant 10026) obtained from EM SCIENCE, a division of EM Industries (Gibbstown, NJ), and Magnesium Test Strips obtained from Environmental Test Systems, Inc. (Elkhart, IN). All chemicals were of reagent grade quality or better and were used without further purification. Distilled-deionized water was used to prepare all solutions. Baker Instra-Analyzed Reagent[R] grade nitric acid (J.T. Baker, Phillipsburg, NJ) was used for metals preservation. Plasma-Pure[R] 1000 mg/L arsenic in 5 per cent nitric acid and 1000 mg/L magnesium in 5 per cent nitric acid stock solutions were purchased from Leeman Labs (Lowell, MA). SAE fine test spark plug dust was obtained from Powder Technology, Inc. (Burnsville, MN). Sea salts were purchased from SIGMA Chemical Company (St. Louis, MO) and Humic acid from Aldrich Chemical Company (Milwaukee, WI). Monacacy River water was filtered through 0.45 mm membrane filters and stored in sterilized containers under refrigeration.

**Materials Preparation.** Chloride stock solution (10,000 mg $Cl^-$/L): 16.485 g sodium chloride (NaCl) were dissolved in water and diluted to 1L. Cyanide stock solution (1,000 mg $CN^-$/L): 2.503 g potassium cyanide (KCN) were dissolved in a solution containing 1.6 g sodium hydroxide (NaOH) and diluted to 1L. All dilutions of this stock solution were made using a solution of 1.6 g NaOH/L. Sulfate stock solution (10,000 mg $SO_4^{-2}$/L): 14.787 g sodium sulfate ($Na_2SO_4$) were dissolved in water and diluted to 1L. Arsenic stock solution (100 mg As/L): 0.133 g arsenic trioxide ($As_2O_3$) were dissolved in 100 mL water containing 0.4 g NaOH. The solution was acidified with 2 mL concentrated nitric acid ($HNO_3$) and diluted to 1L with water. Simulated field water: 1.273 g SAE fine test spark plug dust, 15.08 g sea salts, and 103 mg humic acid were mixed with 1L water. Powdered phosphate buffer consisted of an intimate mixture of 1.36 g potassium dihydrogen phosphate ($KH_2PO_4$) and 1.42 g sodium monohydrogen phosphate ($Na_2HPO_4$).

**Instrumentation.** Concentrations of chloride and sulfate test solutions were confirmed in-house by ion chromatography using a DIONEX 4000i system with an AI 450 auto sampler (Dionex Corporation, Sunnyvale, CA). Concentrations of magnesium and arsenic test solutions were confirmed in-house on an ICAP Model 61E (Thermo Jarrell Ash Corporation, Franklin, MA). Concentrations of cyanide and arsenic test solutions were confirmed by a contract testing lab. High temperature storage evaluations were conducted in a circulating air oven (model 4-3521, American Instrument Company, Silver Spring, MD).

**Experimental Design.** Test strips and procedures were evaluated first by four in-house operators. Three standards for each parameter, one low-level, one mid-level, and one high-level, were prepared by dilution from the appropriate stock standards and analyzed with the corresponding test kit. Evaluations were made as to accuracy, time required to complete the analysis, ease of operation, and clarity of individual instructions.

Military field units were also asked to evaluate the test strip kits. Two solutions, a high and low concentration, for each parameter were prepared from the appropriate stock standards. The pH of these test solutions fell within the range required by the test kits except for the cyanide where the preservation pH differs considerably from that required by the test kit. A powdered phosphate buffer was prepared and a protocol developed for its use to adjust the pH of the cyanide samples to one within the range required for the test kit. A set of the test kits including the additions and instructions that would be sent to the participating field units was used to analyze the test solutions . Concentrations of these solutions were confirmed by instrumental analysis. Test kits, splits of samples to be analyzed, additional kit instructions (Appendix II) and kit performance questionnaires (Appendix III) were sent to four military field units (Appendix IV) for testing by field personnel.

In order to evaluate the test kits' abilities to withstand storage under high temperature extremes, each test kit was held at a temperature of 160 °F, considered the peak temperature developed during storage in hot climates,[4] for a total of 7 days. Starting with the third day, kits were removed from the oven and allowed to cool for one and one-half hours. At this time the kits were examined for physical degradation and for any changes in their analytical capabilities. Kit responses were tested using the same low-level, mid-level, and high-level concentrations of standards used in the initial in-house evaluation studies. Kits were then returned to the oven.

Simulated field water and filtered Monacacy River water were spiked with the individual stock standards to evaluate potential matrix effects. These waters and their spikes were analyzed with the test kits and the spike recoveries determined.

**Statistical Methods.** Analysis of variance was used to test for fit to the model and for effects followed by Duncan's Multiple Range test for comparisons among means. A one-tailed Student's t-test was used to compare test values with instrument values. SAS PROC GLM statistical software was used for ANOVA and Duncan's Multiple Range tests.[5]

## RESULTS

**In-House Evaluations.** Data obtained from in-house analysis of standard solutions with the test kits are summarized in Table 2.

Table 2. Preliminary evaluations of test kits using standard solutions.[a]

| Parameter | Nominal Value | Operator 1 | Operator 2 | Operator 3 | Operator 4 |
|---|---|---|---|---|---|
| Arsenic | 0.1<br>1.0<br>3.0 | 0.1<br>1.0<br>1.7 - 3.0 | 0.1<br>1.0<br>1.7 - 3.0 | 0.1<br>1.0<br>3.0 | 0.1<br>1.0<br>1.7 |
| Magnesium | 4.0<br>12<br>30 | 0<br>10<br>20 | b<br>b<br>b | >0<br>10 - 20<br>20 - 30 | 0<br>20<br>20 |
| Chloride | 500<br>1500<br>3000 | 500<br>1500<br>3000 | 500<br>1500<br>3000 | 500<br>1500<br><3000 | b<br>b<br>b |
| Sulfate | 200<br>800<br>1600 | 200-300<br>800<br>1600 | 200-400<br>800<br>1600 | 200<br>800<br>1600 | 200-400<br>400-800<br>1200-1600 |
| Cyanide | 1.0 c<br>10 c<br>30 c | <1.0<br>10<br><30 | 1.0<br>10<br>30 | 1.0<br>10<br>30 | 1.0<br>10<br>30 |

[a]Concentrations in mg/L.
[b]Test strips were not available for use at the time tests were performed.
[c]Concentrations confirmed by instrumental analysis

All test kits showed acceptable semi-quantitative accuracy. The following general observations were made during the in-house evaluations.

Magnesium, chloride, and sulfate test strips were judged simple and quick to use. However, some ambiguity was noticed in the interpretation of the sulfate test strip responses. Chloride and sulfate test strips are usable over a wide pH range. The magnesium test strips failed in solutions that had been preserved with $HNO_3$ to pH <2.

The cyanide test requires pH adjustment to a narrow range. Reagent 1, a powder, was difficult to dissolve in the sample cup provided without splashing, and reagent 2 gives off a nauseous pyridine odor.

The arsenic test also failed in solutions preserved with $HNO_3$ to a pH <2. The 32 per cent HCl used in the kit is dangerous to handle and the dropping bottle it comes in is difficult to use. Drops are not delivered quickly enough and results can be low because the arsine gas ($AsH_3$) produced at low pH is rapidly lost.

**Field Testing Evaluations.** Military field units asked to participate in this testing are identified in Appendix II. Three out of four units responded. Results of the field testing are given in Table 3.

Participating field units were requested to analyze the solutions 5 times each; once each on 5 different days. Two of the responding units did this. Tests done by field unit 2 were not run on 5 different days as requested and sample ID's were not given for analyses performed by them on 21-22 Jun 92. The assumption was made that the samples analyzed were those with concentrations that agreed most closely with the analyzed values.

All field units reported that test strip instructions were easy to follow, kits easy to use, and kit packaging adequate. One group suggested that it might be advantageous to have individually packaged test strips for in-the-field use.

Proposed changes to the arsenic test kit included replacement of the sample syringe with a pipette and replacement of the 32 per cent HCl delivery bottle either with a plastic squeeze bottle or with an eye dropper assembly. Supplying additional reaction cylinders to enable processing more than one test at a time was also recommended.

Suggested changes to the cyanide test included the addition of a stirring rod and having pH strips and pH adjusting reagents available.

The addition of a color comparator block between the 60 and 120 mg/L blocks was recommended for the magnesium test strips.

It was requested that a supply of deionizer resin and a bottle for deionized water be provided to allow for the rinsing of sample containers and reaction vessels. Keeping the set as compact as possible, that is, one case for all five test kits and any additional material, was also considered important.

Table 3. Results from field unit evaluations.[a]

| Test | Nominal Value | Inst. Value | Field Unit | Run # 1 | Run # 2 | Run # 3 | Run # 4 | Run # 5 | Field Test Dates |
|---|---|---|---|---|---|---|---|---|---|
| As | 0.5 | 0.45 | 1 | 1.0 | 0.8 | 1.0 | 1.0 | 0.8 | 24-30Jun92 |
| | | | 2 | 1.0 | 0.5 | 0.5 | 1.0 | 1.0 | 27Jul92 |
| | | | 3 | 0.1 | 0.5 | 0.5 | 0.5 | 0.5 | 27-31Oct92 |
| As | 1.7 | 1.5 | 1 | 1.7 | 1.7 | 1.7 | 1.7 | 1.7 | 24-30Jun92 |
| | | | 2 | 0 | 1.0 | 1.7 | 1.7 | 1.7 | 21-22Jun92 |
| | | | 2 | 1.7 | 1.7-3.0 | 1.7-3.0 | 1.7-3.0 | 1.7 | 24Jul92 |
| | | | 3 | 1.7 | 1.7 | 1.7 | 1.7 | 1.7 | 27-31Oct92 |
| Mg | 10 | 9.1 | 1 | 10 | 10 | 10 | 10 | 10 | 24-30Jun92 |
| | | | 2 | 15 | 12 | 17 | 17 | 12 | 27Jul92 |
| | | | 3 | 10 | 10 | 10 | 10 | 10 | 27-31Oct92 |
| Mg | 60 | 53.9 | 1 | 120 | 80 | 110 | 120 | 110 | 24-30Jun92 |
| | | | 2 | 120 | 120 | 120 | 120 | 120 | 21-22Jun92 |
| | | | 2 | 60-120 | 60-120 | 60-120 | 60-120 | 60-120 | 24Jul92 |
| | | | 3 | 60 | 60 | 60 | 60 | 60 | 27-31Oct92 |
| $Cl^-$ | 1000 | 1020 | 1 | 1000 | 1500 | 1000 | 1000 | 1000 | 24-30Jun92 |
| | | | 2 | 1500 | 1500 | 1500 | 1500 | 1500 | 21-22Jun92 |
| | | | 2 | 1000 | 1000 | 1000 | 1000 | 1000 | 24Jul92 |
| | | | 2 | 1000 | 1000 | 1000 | 1250 | 1250 | 27Jul92 |
| | | | 3 | 1500 | 1500 | 1500 | 1500 | 1500 | 27-31Oct92 |
| $Cl^-$ | 2500 | 2640 | 1 | 2000 | 2000 | 2000 | 2000 | 2000 | 24-30Jun92 |
| | | | 3 | 2000 | 2000 | 2000 | 2000 | 2000 | 27-31Oct92 |
| $SO_4$= | 800 | 849 | 1 | >800 | >800 | 800 | 800 | 800 | 24-30Jun92 |
| | | | 3 | >800 | >800 | >800 | >800 | >800 | 27-31Oct92 |
| $SO_4$= | 1200 | 1200 | 1 | >1200 | 1200 | 1200 | 1200 | 1200 | 24-30Jun92 |
| | | | 2 | >1200 | >1200 | >1200 | >1200 | >1200 | 21-22Jun92 |
| | | | 2 | >1200 | >1200 | >1200 | >1200 | >1200 | 24Jul92 |
| | | | 2 | 1000 | 900 | 900 | 900 | 900 | 27Jul92 |
| | | | 3 | >1200 | >1200 | >1200 | >1200 | >1200 | 27-31Oct92 |
| $CN^-$ | 1.0 | 1.2 | 1 | 0 | 0 | 0 | 0.5 | 0.5 | 24-30Jun92 |
| | | | 2 | 1 | 0.5 | 1 | 1 | 2 | 27Jul92 |
| | | | 3 | 0.1 | 1 | 1 | 1 | 1 | 27-31Oct92 |
| $CN^-$ | 10 | 13 | 1 | 7 | 7 | 7 | 7 | 7 | 24-30Jun92 |
| | | | 2 | 10 | 10 | 10 | 10 | 10 | 21-22Jul92 |
| | | | 2 | 10 | 3-10 | 3-10 | 3-10 | 3-10 | 24Jul92 |
| | | | 3 | 3 | 3 | 10 | 3 | 3 | 27-31Oct92 |

[a]All values in mg/L.

**Statistical Evaluation of Field Results.** A significant overall unit effect (differences among units) in endpoint measurements was found for As (low concentration), Mg (both concentrations), and $Cl^-$ (low concentration). There were no overall replicate effects for any of the tests and concentrations (Appendix V, tables 1 and 3).

the tests and concentrations (Appendix V, tables 1 and 3).

More detailed analysis for comparisons among units for each test and concentration revealed that for As (low concentration), unit 3 had a significantly lower mean measurement than either of the other two units. For Mg (low concentration), unit 2 was significantly higher than either unit 1 or unit 3, and for Mg (high concentration) unit 1 reported the highest measurements. For $Cl^-$ (low concentration), unit 3 had significantly higher mean measurements than unit 1 or unit 2 (Appendix V, table 2).

Comparisons between pooled means and the instrument value for each test for each concentration indicated that the test measurements for As (low concentration), Mg (both concentrations), and $Cl^-$ (low concentration) were significantly higher than their respective instrument measurements. For $CN^-$, both concentrations had significantly lower test measurements than instrument values. There was no variation in test measurements for As (high concentration), $Cl^-$ (high concentration), and both concentrations of $SO^{4=}$. Table 4 in Appendix V shows these values.

Concentration levels reported for the cyanide samples had the widest variance with known values. Since field unit three analyzed their solutions in October, five months after solution preparation and preservation, and obtained results in complete agreement with the known concentration for the 1.0 mg/L sample, it is deduced that the problem is in use of the kit itself.

**Heated Test Kit Evaluations.** Analytical results obtained with the test kits after being heated are summarized in Table 4.

Table 4. Performance of test strips as a function of high temperature (160°) storage.[a]

| Parameter | Nominal Value | Day 3 | Day 4 | Day 5 | Day 6 | Day 7 |
|---|---|---|---|---|---|---|
| Arsenic | 0.1<br>1.0<br>3.0 | 0.1<br>1.0<br>1.7 | 0.1-0.5<br>1.0<br>1.0-1.7 | 0.5<br>0.5-1.0<br>1.7-3.0 | 0.1-0.5<br>1.0<br>1.7-3.0 | 0.1-0.5<br>1.0-1.7<br>1.7-3.0 |
| Magnesium | 4.0<br>12<br>30 | b<br>b<br>b | b<br>b<br>b | b<br>b<br>b | b<br>b<br>b | b<br>b<br>b |
| Chloride | 500<br>1500<br>3000 | 500<br>1500<br>2000-3000 | 500<br>1500<br>3000 | 500<br>1500<br>3000 | 500<br>1500-2000<br>2000-3000 | 500<br>1500-2000<br>3000 |
| Sulfate | 200<br>800 | 200-300<br>800 | 200<br>800 | 200-400<br>800 | 200-400<br>800 | 200-400<br>800 |
| Cyanide | 1.0<br>10<br>30 | 1.0<br>10<br>30 | 1.0<br>10<br>30 | 1.0<br>10<br>30 | 0-1<br>10[c]<br>10-30[c] | d<br>d<br>d |

[a]All values in mg/L.
[b]Concentrations not ascertained due to change in colors produced. See text.
[c]Center of color block only responding.
[d]Cyanide kit removed from test. See text.

Arsenic Test Kit: As heating progressed, the analyses indicated higher solution concentrations than initially. Kit damage included deformation of the plastic kit container and loosening of the test strip container cover.

Magnesium Test Strips: After the initial days at elevated temperature storage, colors developed on the reaction block for a given concentration of magnesium were lighter and more yellow than those indicated on the comparison card.

Sulfate Test Strips: The cap on the storage container loosened. No apparent deterioration of response of reaction zones was noted, however.

Chloride Test Strips: The cap on the storage container loosened and came off thus exposing the strips to light during the cooling period each day. Because of this, the yellow of the chromate on the test strip was darkened with elemental silver from light induced decomposition of the silver chromate. By the end of the week interpretations of color changes at higher concentrations were questionable.

Cyanide Test Kit: Initial heating caused the storage container to be badly deformed with the reagent cap flattened but still usable. Reagent 1, originally a powder, was now a compacted solid. The cover for the test strip container had loosened. By day 5 a seam on the bottle containing the chloramine T / pyridine solution had given way and there was a pool of the solution in the plastic kit container. The oven reeked of pyridine. The color strips were only registering color development in the center of the color blocks. Day six found pyridine solution all over the kit box and the buffer bottle. Colors of the comparison chart were deteriorating. This kit was removed from any further heat testing.

**Interference Evaluations.** Results from the spike analysis of simulated field water and Monacacy River water are summarized in Table 5.

Sulfate test strips registered false positives throughout the range of unspiked and spiked samples for both simulated field water and Monacacy River water.

Monacacy River water exerted a negative interference on cyanide test kit analysis but a positive interference on instrumental analysis.

Table 5a. Interference data from simulated field water analysis.[a,b,c]

| | Sample Analysis | | | Spike Analysis | |
|---|---|---|---|---|---|
| Parameter | Inst. Value | Test Strip Value | Spike Added | Inst. Value | Test Strip Value |
| Arsenic | 0.012 | <0.1 | 0.5 | 0.656 | 0.5 |
| Magnesium | 47.3 | 40 | 20 | 67.9 | 60 |
| Sulfate | 97 | 200-400 | 200 | 300 | >400 |
| Cyanide | <0.01 | <1 | 3 | 3.5 | 3 |

Table 5b. Interference data from Monacacy River water analysis.[a,b,c]

| | Sample Analysis | | | Spike Analysis | |
|---|---|---|---|---|---|
| Parameter | Inst. Value | Test Strip Value | Spike Added | Inst. Value | Test Strip Value |
| Arsenic | 0.047 | <0.1 | 0.5 | 0.692 | 0.5 |
| Magnesium | 7.85 | 10 | 20 | 29.6 | 30 |
| Sulfate | 23 | 200-400 | 200 | 215 | >400 |
| Cyanide | <0.01 | <1 | 3 | 4.8 | <1 |

[a]Chloride test strips not available at time of testing
[b]All analyses on this table were performed in-house except for the cyanide
[c]All values in mg/L

## DISCUSSION

As indicated on Table 1, current detection ranges for test strip technology are fairly close to those proposed as being necessary for field preventive medicine units. When evaluating the test strip results obtained by the field units, the pooled values (Appendix V, Table 4) are significantly different from those obtained instrumentally. However, when these values are compared with the proposed detection ranges, they do appear to provide useful information which could be valuable to preventive medicine units under field conditions.

Inexperienced operators should run some practice tests to become used to the procedures and equipment involved with the different kits, especially in the case of the arsenic and cyanide kits. Timing and finesse are important to the success of both these tests.

The pH of waters that field units are likely to encounter can be accomnmodated by all the tests with the exception of the cyanide test. This test requires a sample of pH between six and seven. To this end, the analysis set must include pH test strips of some type and reagents to adjust both acidic and alkaline waters.

Small stirring rods should be included in the set to ensure complete mixing of sample with reagents for any pH adjustments needed prior to testing. Stir rods also would be helpful in the cyanide test to ensure complete dissolution of reagent 1, a powdered buffer.

Standard operating procedures for cleaning sample containers and reaction vessels need to be developed. Rinsing sample containers and reaction vessels at least four times with the water to be analyzed should be sufficient. In order to keep the size of the analysis set as small as possible, inclusion of deionizer resin and bottles for deionized water or the carrying of supplies of distilled water is not recommended.

Storage conditions for these kits need to be addressed. All kits have a 24 month shelf life from the time of manufacture[6,7,8]. EM Science recommends that their kits be stored under refrigeration until opened. Once opened they should be stored at room temperature in a dry area. The caps for the strip containers contain a silica plug especially designed to keep these strips dry. The silica plugs are ruined when subjected to moisture. Also, kit responses cannot be guaranteed if kits are stored under desert temperatures[6]. The magnesium test strips are to be stored at room temperature[7].

The evaluations of the kits at elevated temperatures showed that test strips in general need to be protected from drying out. The manufacturer has made use of protective packaging to protect the strips from excess moisture. Therefore it is highly recommended that test strips for use under field conditions be individually packaged to prevent their drying out or their contact with excess moisture.

The cyanide kit presents unique problems if stored at elevated temperatures. Its chloramine T / pyridine solution developed enough pressure when warmed that the seam of the container split releasing its contents into the kit container. This not only compromised the reagent, but gave off noxious vapors of pyridine.

Upon being heated to 160 °F (71.1 °C) the color developed by the magnesium strips changed character. A technical advisor for Environmental Test Systems, Inc. suggests that a discoloration of the paper matrix occurs at that temperature. A temperature no higher than 60 °C was used in their heat stress testing of the strips and they saw no significant change in the test strips[6].

## CONCLUSION

Off-the-shelf test strip kit technology currently available for arsenic, magnesium, chloride, sulfate, and cyanide could serve as a basis for an interim WQAS-PM with some minor adjustments and additions. All test kits showed semi-quantitative accuracy during in-house testing by experienced personnel, and they appear to provide useful information on water quality even when used by inexperienced field units.

Further developmental work needs to be done to extend the detection ranges for some of the test strips and extend the shelf life for all of them.

This prototype WQAS-PM represents an interim fix toward fulfillment of the Army's requirements for evaluating potability of field waters.

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5. SAS Institute Inc., SAS / STAT ™ User's Guide Release 6.03 Edition. Cary, NC: SAS Institute Inc., 1988. 1028pp

6. Telephone conversation with Al Sutherland, EM Science, 2 Feb 93

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## APPENDIX I

### Test Kit Descriptions and Procedures

**Chloride Test Kit (Merckoquant 10079):** Kit consists of test strips for the detection and semi-quantitative determination of chloride ions. Five reaction zones consisting of varying amounts of red-brown silver chromate are sealed on the strips. The chloride ions react with the silver leaving the yellow chromate. The concentration of chloride is measured by visual comparison of the reaction zones of the test strip with the individual color rows of a color scale. The measuring range /color scale for this test is 500 - 1000 - 1500 - 2000 - 3000 mg/L chloride.

Use of the kit involves the following: (1) the pH of the sample solution must be within the range 5-8. Adjust, if necessary, with sodium hydroxide solution or nitric acid; (2) remove test strip and **immediately reclose container**; (3) immerse test strip **with all reaction zones** briefly (approximately 1 sec) in sample solution; (4) gently shake off excess liquid from test strip; and (5) after 1 minute compare color pattern of the reaction zones with the color rows on label. Determine with which row the color pattern coincides and read off corresponding concentration value. If an exact color match cannot be achieved, estimate an intermediate value.

**Sulfate Test Kit (Merckoquant 10019).** Kit consists of test strips for the detection and semi-quantitative determination of sulfate ions. There are four test zones containing varying amounts of the red-colored thorin-barium complex sealed onto plastic strips. In the presence of the equivalent amounts of sulfate ions, the complex is broken up with the removal of the barium leaving the yellow thorin.

Use of the kit involves the following: (1) the pH value of the test solution should be 4-8. Acidic solutions (pH<4) are to be buffered with sodium acetate. Alkaline solutions (pH>8) are adjusted by the addition of ascorbic acid or tartaric acid; (2) immerse the test strip briefly in the test solution until all four zones are completely wetted; and (3) wipe excess liquid off on the edge of the vessel and assess the coloration after 2 minutes.

Assessment:

| | | |
|---|---|---|
| 4 zones light red: | less than | 200 mg/L sulfate |
| 3 zones light red, 1 zone yellow: | over | 400 mg/L sulfate |
| 2 zones light red, 2 zones yellow: | over | 800 mg/L sulfate |
| 1 zone light red, 3 zones yellow: | over | 1200 mg/L sulfate |
| 4 zones yellow: | over | 1600 mg/L sulfate |

The four test zones change from light red to yellow at various sulfate concentrations.

| Transition interval: | Zone: |
|---|---|
| 300 - 400 mg/L | zone first wetted |
| 600 - 800 mg/L | |
| 1000 -1200 mg/L | subsequent zones |
| 1300 - 600 mg/L | |

If the test solution has a sulfate content just at the upper limit of one transition interval, the respective test zone exhibits an approximately 4 mm-wide yellow band which still has red edges on both sides. At higher contents the edges also change to yellow.

**Cyanide Test Kit (Merckoquant 10044):** Kit consists of test strips and reagents for the detection and semi-quantitative determination of dissociable (freely released) cyanides. Reagent 1 is a

powered buffer for optimizing the pH of the reaction medium. Reagent 2 is a liquid containing chloramine T, a source of chlorine, in pyridine solution. Cyanide ions react with chlorine to form cyanogen chloride which opens the pyridine ring giving glutaconic dialdehyde. The barbituric acid on the test zone of the strip reacts with the dialdehyde and produces a red polymethine dye. The measuring range / color scale for the test is 0 - 1 - 3 - 10 - 30 mg/L cyanide.

Use of the kit involves the following: (1) if the water is strongly acidic or alkaline, pH of the water to be tested must be adjusted to between 6 and 7; (2) rinse the measuring vessel several times with the solution to be tested and fill to the 5-mL mark; (3) determine the pH value; (4) if the pH is greater than 9, add 25 per cent sulfuric acid dropwise until the pH is between 6 and 7; (5) add 1 level dosing spoon of reagent 1 and dissolve; (6) add 5 drops of reagent 2 and shake well; (7) immediately immerse the reaction zone of the test strip in the solution for 30 seconds; and (8) remove the test strip, shake off excess liquid and compare the reaction zone with the color scale within 10 seconds.

**Arsenic (Merckoquant 10026):** The kit consists of test strips, reagents and accessories for the detection and semiquantitative determination of tri- and pentavalent inorganic arsenic compounds. Inorganic arsenic compounds in the solution to be tested are converted to arsine by adding zinc and hydrochloric acid. The test strip reaction zone containing mercury(II) bromide is suspended in the head space above the test solution. The arsine reacts with the mercury(II) bromide to form mixed arsenic mercury halogenides giving a yellow to brown coloration. The measuring range / color scale for this test is 0.1 - 0.5 - 1.0 - 1.7- 3.0 mg/L arsenic.

Use of the kit involves the following: (1) insert a test strip halfway through the slot in the cap of the reaction vessel, reaction zone first; (2) transfer 5 mL of the solution to be tested to the reaction vessel using the syringe, add one measuring spoonful of Reagent 1 (zinc) and shake; (3) add 10 drops of Reagent 2 (32 per cent HCl) and immediately close the reagent vessel with the cap; (4) leave to react for 30 minutes, gently swirling 2 or 3 times; and (5) remove the test strip, briefly dip into water, gently shake off and compare the reaction zone with the color scale.

**Magnesium Test Strips.** The test strips consist of one reaction zone sealed on to plastic strips for the semiquantitative determination of magnesium in solution. Use of the strips involves the following: (1) dip strip in sample for 3 seconds; (2) remove strip; (3) read immediately at 30 seconds; and (4) interpolate between color blocks if necessary. The range / color scale for this test is 0 - 10 - 20 - 30 - 60 - 120 mg/L magnesium.

## APPENDIX II

### WQAS-PM Test Methods Addendum

The cyanide samples have been preserved to maintain stability. This preservation leaves these samples at a pH outside the recommended range for successful testing with the kit. Included in this Water Quality Analysis Set is a bottle of phosphate buffer along with a small black plastic scoop. This buffer is to be used for pH adjustment of the cyanide sample.

Cyanide Test: Measure five ml sample into test cup. Add two scoops buffer and dissolve. Proceed with test according to instructions.

Included in this set is an extra cup. This is to be used when performing the magnesium, chloride and sulfate tests. Pour a small portion of sample into the cup and dip the test strip into this sample portion. Rinse the cup with distilled water between tests.

## APPENDIX III

### Questionnaire Sent to Participating Military Field Units

### TEST STRIP QUESTIONNAIRE

1. Are you colorblind? ____yes ____no
2. Are test strip instructions easy to follow? ____yes ____no
3. Are the test strips easy to use? ____yes ____no
4. Is test strip packaging acceptable? ____yes ____no
5. Should test strips be individually packaged? ____yes ____no
6. What changes do you suggest to this water quality analysis set?

7. Other Comments:

## APPENDIX IV

### Participating Military Field Units

1. Academy of Health Sciences, United States Army, Fort Sam Houston, Texas 78234-6100
2. U.S. Army Special Operations Command, 7th Special Forces Group Medical Section, Fort Bragg, North Carolina 28307-5270
3. Navy Environmental and Preventive Medicine Unit No. Two, Microbiology Department, Norfolk, Virginia 23511
4. 5th Preventive Medicine Unit, Unit #15247, APO AP96205-0020

## APPENDIX V

### Statistical Evaluation Tables

Table 1. Effects by test and by concentration.

| Test | Unit | | Replicate | |
|---|---|---|---|---|
| Concentration[a] | F | P | F | P |
| As | | | | |
| 0.45 | 7.20 | 0.0163 | 0.51 | 0.7281 |
| 1.5 | *[b] | * | * | * |
| Mg | | | | |
| 9.1 | 16.79 | 0.0014 | 1.00 | 0.4609 |
| 52.1 | 42.67 | 0.0001 | 1.00 | 0.4609 |
| $Cl^-$ | | | | |
| 1020 | 21.00 | 0.0007 | 1.00 | 0.4609 |
| 2640 | * | * | * | * |
| $SO_4^=$ | | | | |
| 849 | * | * | * | * |
| 1200 | * | * | * | * |
| $CN^-$ | | | | |
| 1.2 | 2.76 | 0.1225 | 0.83 | 0.5404 |
| 13 | 1.53 | 0.2732 | 0.67 | 0.6328 |

$\alpha = 0.05$

[a]All values in mg/L

[b]No error in measured values

## Appendix V

## Statistical Evaluation Tables

Table 2. Comparisons among unit means by test and by concentration.[a]

| Test concentration/ Unit | Mean | N | +/- SD | Duncan Grouping[b] |
|---|---|---|---|---|
| As (0.45) | | | | |
| Unit 1 | 0.92 | 5 | 0.109 | A |
| Unit 2 | 0.80 | 5 | 0.273 | A |
| Unit 3 | 0.42 | 5 | 0.178 | B |
| As (1.5) | | | | |
| Unit 1 | 1.70 | 5 | 0 | A |
| Unit 2 | 1.70 | 5 | 0 | A |
| Unit 3 | 1.70 | 5 | 0 | A |
| Mg (9.1) | | | | |
| Unit 1 | 10.00 | 5 | 0 | B |
| Unit 2 | 14.60 | 5 | 2.51 | A |
| Unit 3 | 10.00 | 5 | 0 | B |
| Mg (53.9) | | | | |
| Unit 1 | 108.00 | 5 | 16.43 | A |
| Unit 2 | 60.00 | 5 | 0 | B |
| Unit 3 | 60.00 | 5 | 0 | B |
| $Cl^-$ (1020) | | | | |
| Unit 1 | 1100 | 5 | 223.6 | B |
| Unit 2 | 1000 | 5 | 0 | B |
| Unit 3 | 1500 | 5 | 0 | A |
| $Cl^-$ (2640) | | | | |
| Unit 1 | 2000 | 5 | 0 | A |
| Unit 2 | no response | | | |
| Unit 3 | 2000 | 5 | 0 | A |
| $SO_4^=$ (849) | | | | |
| Unit 1 | 800 | 5 | 0 | A |
| Unit 2 | no response | | | |
| Unit 3 | 800 | 5 | 0 | A |
| $SO_4^=$ (1200) | | | | |
| Unit 1 | 1200 | 5 | 0 | A |
| Unit 2 | 1200 | 5 | 0 | A |
| Unit 3 | 1200 | 5 | 0 | A |
| $CN^-$ (1.2) | | | | |
| Unit 1 | 0.40 | 5 | 0.41 | A |
| Unit 2 | 1.10 | 5 | 0.54 | A |
| Unit 3 | 0.82 | 5 | 0.40 | A |
| $CN^-$ (13) | | | | |
| Unit 1 | 7.0 | 5 | 0 | A |
| Unit 2 | 4.4 | 5 | 3.13 | A |
| Unit 3 | 4.4 | 5 | 3.13 | A |

[a]All values in mg/L.

[b]Duncan's Multiple Pair Test; $\alpha = 0.05$ for each pairwise comparison. (Means with different letters are different from each other.)

## Appendix V

## Statistical Evaluation Tables

Table 3. Comparisons among replicate means by test and by concentration.[a]

| Test Concentration | Mean | N | +/- SD | Duncan[b] Grouping |
|---|---|---|---|---|
| As (0.45) | | | | |
| 1 | 0.70 | 3 | 0.519 | A |
| 2 | 0.60 | 3 | 0.173 | A |
| 3 | 0.66 | 3 | 0.288 | A |
| 4 | 0.83 | 3 | 0.288 | A |
| 5 | 0.76 | 3 | 0.251 | A |
| As (1.5) | | | | |
| 1 | 1.70 | 3 | 0 | A |
| 2 | 1.70 | 3 | 0 | A |
| 3 | 1.70 | 3 | 0 | A |
| 4 | 1.70 | 3 | 0 | A |
| 5 | 1.70 | 3 | 0 | A |
| Mg (9.1) | | | | |
| 1 | 11.66 | 3 | 2.88 | A |
| 2 | 10.66 | 3 | 1.15 | A |
| 3 | 12.33 | 3 | 4.04 | A |
| 4 | 12.33 | 3 | 4.04 | A |
| 5 | 10.66 | 3 | 1.15 | A |
| Mg (53.9) | | | | |
| 1 | 80.00 | 3 | 34.64 | A |
| 2 | 66.66 | 3 | 11.54 | A |
| 3 | 76.66 | 3 | 28.86 | A |
| 4 | 80.00 | 3 | | A |
| 5 | 76.66 | 3 | | A |
| $Cl^-$(1020) | | | | |
| 1 | 1166.7 | 3 | 288.68 | A |
| 2 | 1333.3 | 3 | 288.68 | A |
| 3 | 1166.7 | 3 | 288.68 | A |
| 4 | 1166.7 | 3 | 288.68 | A |
| 5 | 1166.7 | 3 | 288.68 | A |
| $Cl^-$(2640) | | | | |
| 1 | 2000 | 2 | 0 | A |
| 2 | 2000 | 2 | 0 | A |
| 3 | 2000 | 2 | 0 | A |
| 4 | 2000 | 2 | 0 | A |
| 5 | 2000 | 2 | 0 | A |

## Appendix V

## Statistical Evaluation Tables

Table 3. Cont'd.

| Test Concentration[a] | Mean | N | +/- SD | Duncan[b] Grouping |
|---|---|---|---|---|
| $SO_4^{=}$(849) | | | | |
| 1 | 800 | 2 | 0 | A |
| 2 | 800 | 2 | 0 | A |
| 3 | 800 | 2 | 0 | A |
| 4 | 800 | 2 | 0 | A |
| 5 | 800 | 2 | 0 | A |
| $SO_4^{=}$(1200) | | | | |
| 1 | 1200 | 3 | 0 | A |
| 2 | 1200 | 3 | 0 | A |
| 3 | 1200 | 3 | 0 | A |
| 4 | 1200 | 3 | 0 | A |
| 5 | 1200 | 3 | 0 | A |
| $CN^{-}$(1.2) | | | | |
| 1 | 0.70 | 3 | 0.520 | A |
| 2 | 0.50 | 3 | 0.500 | A |
| 3 | 0.67 | 3 | 0.577 | A |
| 4 | 0.83 | 3 | 0.287 | A |
| 5 | 1.17 | 3 | 0.764 | A |
| $CN^{-}$(13) | | | | |
| 1 | 6.7 | 3 | 3.512 | A |
| 2 | 4.3 | 3 | 2.309 | A |
| 3 | 6.7 | 3 | 3.512 | A |
| 4 | 4.3 | 3 | 2.309 | A |
| 5 | 4.3 | 3 | 2.309 | A |

[a]All values in mg/L

[b]Duncan's Multiple Range Test; a = 0.05 for each pairwise comparison. (Means with different letters are different from each other.)

## Appendix V

### Statistical Evaluation Tables

Table 4. Comparisons between pooled means and instrument values for each test and for each concentration.[a]

| Test | Inst. Value | Test Value (Mean) | +/- SE | N | t | p |
|---|---|---|---|---|---|---|
| As | 0.45<br>1.5 | 0.71<br>1.7 | 0.074<br>0 | 15<br>15 | 3.547<br>NE | <0.05<br>NE |
| Mg | 9.1<br>53.9 | 11.53<br>76.0 | 0.675<br>6.459 | 15<br>15 | 3.604<br>3.422 | <0.05<br><0.05 |
| $Cl^-$ | 1020<br>2640 | 1200<br>2000 | 65.47<br>0 | 15<br>10 | 2.750<br>NE | <0.05<br>NE |
| $SO_4^{-2}$ | 849<br>1200 | 800<br>1200 | 0<br>0 | 15<br>15 | NE<br>NE | NE<br>NE |
| $CN^-$ | 1.2<br>13 | 0.773<br>5.27 | 0.134<br>0.693 | 15<br>15 | 3.179<br>11.155 | <0.05<br><0.05 |

[a]All values in mg/L.

$\alpha = 0.05$. One-tailed Student's t-test

NE = No error on measured values.

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