Appendix A – Joint Memorandum
TO: ALL MEMBERS OF THE DEPARTMENT

A team has been established to fully evaluate the three recent incidents leading up to the cyanide exposures sustained by members.

The team will be comprised of the following:

Deputy Assistant Chief J. Curtis Varone
Battalion Chief Thomas N. Warren
Lieutenant Kevin L. Jutras
Firefighter Joseph L. Molia, Health and Safety Representative for Local 799
Arson Investigator Joseph F. Dorsey

The evaluation process has begun and firefighters that were involved with these incidents should be expected to be contacted to be interviewed.

The objectives of the team evaluation are:

1. To determine the direct and indirect causal factors which resulted in the exposure of several firefighters to cyanide and the life threatening situation that resulted to one of our firefighters, particularly those factors that could be used to prevent future occurrences of a similar nature, including:
   a. Identifying inadequacies involving apparatus, equipment, protective clothing, standard operating procedures, supervision, training, or performance.
   b. Identifying situations that involve an unacceptable risk.
   c. Identifying previously unknown or unanticipated hazards

2. To ensure that the lessons learned from the investigation are effectively communicated to prevent future occurrences of a similar nature.

3. To ensure that the incident and all related events are fully documented and evidence is preserved.
4. To provide factual information to assist those involved who are trying to understand the events they experienced.

5. To provide the information to other individuals and organizations that is involved in the cause of fire service occupational safety and health.

6. The mission of the team is to find facts and develop recommendations for changes in equipment, training, or procedures in order to prevent similar incidents. It is not a mechanism to investigate or assess blame, or to lead to discipline.

DAVID D. COSTA  \hspace{5cm}  PAUL A. DOUGHTY
Chief of Department  \hspace{5cm}  President, Local 799
Appendix B – Optic Neuritis Info
September 14, 1990

Dr. Simmons Lessell
Mass Eye & Ear
243 Charles Street
Boston, MA 02114

Dear Dr. Lessell;

On behalf of the Providence Firefighters Local 799 of the International Association of Firefighters, I want to thank you for your recent assistance provided to the four members of our Local who experienced vision loss in their left eye. I believe you have permitted each of the four members involved, as well as the other 479 members of the Providence Fire Department, to rest a little easier under the circumstances.

I do have several concerns, however, which I was unable to discuss with you when we met on August 28, 1990. I would appreciate it if you could find the time to consider these questions so that we may put this entire episode behind us.

First of all, the medical reports of each of the four patients indicates that either they do, or do not, have a form of optic neuritis. However, the reports do not discuss whether or not a common exposure could have caused papillitis in one patient, retrobulbar neuritis in another patient, and retinal vein occlusion in yet another. Certainly it would not be unheard of for two people to be exposed to the same disease or chemical, and develop strikingly different symptoms depending upon a multitude of factors such as their age, physical condition, immune status, pre-existing diseases, length and type of exposure, etc. This possibility remains our primary concern more so than the correctness of a specific diagnosis.

As you may well imagine, firefighters are routinely exposed to numerous types of chemicals on a daily basis, as well as being exposed to just about every type of bacteria, fungus or virus known to man on rescue runs. All four of the affected members were assigned to the same shift, which means that they responded to many of the same incidents. There are approximately 120 firefighters per shift in Providence.

Furthermore, the onset of all four cases was within a relatively short period. All four are the same rank (Firefighter
1st Class, as opposed to Lieutenants, Captains or Chiefs). All four were assigned to the fire force, as oppose to rescue, fire prevention, fire alarm, etc. There are roughly 70 fire force firefighters per shift. Thus four firefighters out of a pool of 70 firefighters working on the same shift experienced vision loss in their left eye at approximately the same time. No other firefighter on any other shift has similarly experienced such a problem with either eye.

What limited research we have been able to do on our own to date has not been particularly enlightening. However, two items stand out that I think need to be addressed before we close the file on this matter once and for all. The first is the question of Sarcoidosis. Enclosed in an article regarding the manifestation of sarcoidosis in the eye. This article leaves no doubt that Sarcoidosis may initially (or only) present as "Papillitis", "Retrobulbar neuritis", or "Retinal perivasculitis" to mention a few. While admittedly, this may seem a little (or a lot) far fetched, let me set the stage for you.

At the present time five members of the Providence Fire Department have been diagnosed as having Sarcoidosis. Three of these have been confirmed by biopsy, the other two by clinical symptoms. The cluster of sarcoid cases appears to be associated with a particular exposure that occurred while the affected members were in training. As of yet the exact cause for the cluster has not been determined, although several leads are presently being followed up by Dr. David Kern, with whom you are acquainted.

While none of the four patients you examined fits the profile of the five sarcoidosis cases we have, I believe Sarcoidosis should be considered as a possible cause, and if appropriate, ruled out. To me it would seem no less surprising that sarcoidosis is the cause of these patients' vision problems, than it would that five other members would develop sarcoidosis in the first place. If fact, if an infectious agent is in fact found to be the cause of our sarcoidosis cluster, it could explain the eye problem.

The other concern we would like you to consider arose when we tried to hypothesize a mechanism of exposure that would: 1. expose the left eye more often than the right eye; 2. explain why firefighters but not officers were affected; 3. explain why fire force firefighters but not rescue, fire prevention or fire alarm personnel were affected; and 4. explain why only one shift out of four was affected. In doing so we were able to come up with a possible scenario using the parasite Toxocara canis as one possible causative agent that meets three of the four conditions.

The scenario would be of a fire that all four members responded to during the normal course of their shift. The fire involved a house where dogs infected with Toxocara canis resided. Fire hoses were dragged through the house during the routine
course of fighting the fire at which time they became contaminat-
ed with Toxocara canis containing feces. After the fire was
extinguished, the hose was packed by hand back on the apparatus
by the affected members. Officers usually do no engage in this
activity, only firefighters. The affected firefighters thereaf-
ter introduced the parasite into their systems before they were
able to clean up.

I use the Toxocara canis parasite only as one possible
causative agent that we believe should be considered. Perhaps
you know of other diseases or chemicals capable of causing such
eye problems that would better fit our scenario.

While this may seem like a relatively obvious and
routine set of cases to you, I assure you it is not at all obvi-
ous and routine for our members and their families. I know I
speak on behalf of all 479 members of Local 799 and their fami-
lies when I say that your assistance and guidance in this matter
would be greatly appreciated. The general mood on the job is one
of concern despite your assurance to me that the cases did not
appear to be related. Whatever additional information and guid-
ance you can provide to us would, again, be greatly appreciated.

Very truly yours;

J. Curtis Varone, Esq.
Lieutenant, Prov. F.D.
Health & Safety Rep.
Local 799, IAFF
J. Curtis Varone  
Attorney at Law  
11 Central Avenue  
North Providence, RI 02911

Dear Mr. Varone:

Thank you very much for your letter of September 14, 1990. I reviewed it very carefully. Let me be specific about the diagnoses that were made. [REDACTED] had optic neuritis and the cause is not clear. There is nothing in the history to suggest that this has a toxic basis or that it reflects any underlying disease. [REDACTED] also appears to have optic neuritis and in his case there is also no evidence from the history that the problem was the consequence of any environmental or occupational exposure to a toxic agent or infectious organism. It is notable that these two individuals had onset at around the same time but otherwise, I see no reason to conclude that this is more than a chance occurrence. [REDACTED] has a central retinal vein occlusion of unknown cause and does not, or more exactly did not, have optic neuritis. There is no reason to conclude that his central vein occlusion was the consequence of any exposure to environmental or occupational toxins or infectious agents. His problem occurred in a setting of hypertension and diabetes which are known to predispose to central vein occlusions and they are the putative cause in his case. [REDACTED] has a maculopathy which is not optic neuritis and, in fact, involves the retina. There is no evidence it is a toxic or infectious problem.

It is impossible for me to identify a common theme among these four cases, and I cannot identify an etiologic agent that would be manifest in these patients in such disparate ways. There is no ophthalmic evidence that any of these patients have sarcoidosis.

I appreciate the concern that the members and families have but I cannot find an occupational basis for the problem and therefore can't identify measures that might protect other individuals from similar involvement.

Sincerely,

Simmons Lessell, M.D.
Appendix C – Follow Up Survey
Appendix D - Trace Analytics Lab Results
Sampled for Providence F.D. Air Supply One


<table>
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<th>Limiting Characteristic</th>
<th>Concentration</th>
<th>QC Results, %*</th>
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<td>Methane (CH₄), ppmv</td>
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<td>TVHC (excluding CH₄), ppmv</td>
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<td>Odor (provided by customer)</td>
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This sample COMPLIES with the above referenced specification.

Customer Comments:

TRACES

(W) Dew point is expressed in °F at one atmosphere pressure absolute.

*Accuracy relates observed to expected results (100% is complete agreement). Precision relates to reproducibility (0.0% is complete agreement).
Shipman's Fire Equipment Co., Inc.
Mr. Joe Martin
PO Box 257
Waterford CT 06385-0257

Date Received: Tue, March 28, 2006
Date Analyzed: Tue, March 28, 2006
Date Reported: Tue, March 28, 2006
Sampled By: Robert Warren
Date Sampled: Sat, March 25, 2006
Air Source ID: Eagle Storage Banks

Sampled for Providence F.D.


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This sample COMPLIES with the above referenced specification.

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Customer Comments

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(W) Dew point is expressed in ºF at one atmosphere pressure absolute.

---

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Trace Analytics, Inc.
15768 Hamilton Pool Road • Austin, Texas 78738
Voice: 800-AIR-1024 or 512-263-0000 • Fax: 512-263-0002
E-mail: Service@AirCheckLab.com

AirCheck Report
Copyright© 2006 Trace Analytics, Inc.

Customer No.: 1540
Report No.: 05-22027

Hope Air Systems
Ms. Melissa Waskiewicz
PO Box 840
Northboro MA 01532

Date Received: Thu, December 29, 2005
Date Analyzed: Tue, January 3, 2006
Date Reported: Wed, January 4, 2006
Sampled By: Mark Tesson
Date Sampled: Thu, December 15, 2005
Air Source ID: Model RAO15G3C4E, S/N 53526101, Air Supply #2 Truck

Sampled for Providence Fire Dept.
Results vs NFPA 1500-2002 & CGA G-7.1-2004 Grade D Gas Quality Specification

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<td>20.9</td>
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<tr>
<td>Nitrogen / Argon, Volume %</td>
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<td>78.2 / 0.9</td>
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This sample COMPLIES with the above referenced specification.

Customer Comments

TNK Trace

(W) Dew point is expressed in °F at one atmosphere pressure absolute.

*Accuracy relates observed to expected results (100% is complete agreement). Precision relates to reproducibility (0.0% is complete agreement).

Analysis:
Gases & Vapors CAT-A-01 Gas Chromatography/Mass Spectrometry
Oil & Particulate CAT-A-03 Analytical gravimetry
Particle Size CAT-A-04 Optical Microscopy

Source Bottle: 719590
Sample: 117831
Ambient Bottle: 406547

Accredited Since 1991 By
American Association for Laboratory Accreditation
A2LA Certificate No. 322.01

Richard A. Smith, C.I.H., Laboratory Director
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Sampled for Providence Fire Dept.

Results vs NFPA 1500-2002 & CGA G-7.1-2004 Grade D Gas Quality Specification

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This sample COMPLIES with the above referenced specification.

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Customer Comments:

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*Accuracy relates observed to expected results (100% is complete agreement). Precision relates to reproducibility (0.0% is complete agreement).

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Truescale Laboratories

American Association for Laboratory Accreditation

Accredited Since 1991

Richard A. Smith, C.L.H., Laboratory Director

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<th>Specification</th>
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This sample COMPLIES with the above referenced specification.

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**Customer Comments**

(W) Dew point is expressed in °F at one atmosphere pressure absolute.

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**TNOTES**

*Accuracy relates observed to expected results (100% is complete agreement). Precision relates to reproducibility (0.0% is complete agreement).**

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AZLA Certificate No. 322.01
Accredited In The Chemical Field of Testing

Richard A. Smith, C.I.H., Laboratory Director

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Shipman's Fire Equipment Co., Inc.
Mr. Joe Martin
PO Box 257
Waterford CT 06385-0257

Sampled for Providence F.D. Air Supply 1

Results vs NFPA 1989-2003 Gas Quality Specification

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<td>1.8</td>
</tr>
<tr>
<td></td>
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<td>None/Slight</td>
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<tr>
<td>Other</td>
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</table>

This sample COMPLIES with the above referenced specification.

Customer Comments

TNOC

(W) Dew point is expressed in °F at one atmosphere pressure absolute.

*Accuracy relates observed to expected results (100% is complete agreement). Precision relates to reproducibility (0.0% is complete agreement).

American Association for Laboratory Accreditation
AZLA Certificate No. 322-01

Results relate only to items tested. This report shall not be reproduced except in full without the written permission of Trace Analytics, Inc.
Trace Analytics, Inc.
CERTIFIES THAT

Providence Fire Dept.
is in compliance with the compressed gas specification as described by

NFPA 1500-2002 & CGA G-7.1-2004 Grade D

for a sample described as from the compressed gas source

Model RA015G3C4E, S/N 63526101, Air Supply #2 Truck

analyzed on 1/3/06 as documented in Report No. 05-22027.

[Signature]
Richard A. Smith, C.I.H. Laboratory Director

DATE LAST SAMPLED: 12/19/2005
SAMPLING SCHEDULE: Semi-Annual
THE NEXT SAMPLE IS DUE APPROXIMATELY
6/15/2006

LABORATORY ACCREDITED BY THE AMERICAN ASSOCIATION FOR LABORATORY ACCREDITATION
IN THE CHEMICAL FIELD OF TESTING
RESULTS REPORTED RELATE ONLY TO THE ITEMS TESTED.
CERTIFICATE AND REPORT SHALL NOT BE REPRODUCED EXCEPT IN FULL WITHOUT THE WRITTEN PERMISSION OF THIS LABORATORY.
Trace Analytics, Inc.
CERTIFIES THAT

Providence Fire Dept.
is in compliance with the compressed gas specification as described by
NFPA 1500-2002 & CGA G-7.1-2004 Grade D
for a sample described as from the compressed gas source
Model BAP15TH3, S/N 42050101
analyzed on 1/3/06 as documented in Report No. 05-22029.

Richard A. Smith, C.I.H. Laboratory Director

DATE LAST SAMPLED: 12/19/2005
SAMPLING SCHEDULE: Semi-Annual
THE NEXT SAMPLE IS DUE APPROXIMATELY
6/15/2006

LABORATORY ACCREDITED BY THE AMERICAN ASSOCIATION FOR LABORATORY ACCREDITATION
IN THE CHEMICAL FIELD OF TESTING
RESULTS REPORTED RELATE ONLY TO THE ITEMS TESTED.
CERTIFICATE AND REPORT SHALL NOT BE REPRODUCED EXCEPT IN FULL WITHOUT THE WRITTEN PERMISSION OF THIS LABORATORY.
Trace Analytics, Inc.

CERTIFIES THAT

Providence F.D.

is in compliance with the compressed breathing air specification as described by

NFPA 1500-2002 & CGA G-7.1-2004 Grade E

for a sample described as from the compressed gas source

IR, S/N: 966574

analyzed on _____1/2/06____ as documented in Report No. _____05-21984____

DATE LAST SAMPLED: 12/20/2005
SAMPLING SCHEDULE: Quarterly
YOUR NEXT SAMPLE IS DUE:

3/20/2006

Richard A. Smith, C.I.H., Laboratory Director

Trace Analytics, Inc.
15768 Hamilton Pool Rd.
Austin, TX 78738 800-AIR-1024

LABORATORY ACCREDITED BY THE AMERICAN ASSOCIATION FOR LABORATORY ACCREDITATION
IN THE CHEMICAL FIELD OF TESTING
RESULTS REPORTED RELATE ONLY TO THE ITEMS TESTED.
CERTIFICATE AND REPORT SHALL NOT BE REPRODUCED EXCEPT IN FULL WITHOUT THE WRITTEN PERMISSION OF THIS LABORATORY.
Trace Analytics, Inc.

CERTIFIES THAT

Providence F.D. Air Supply 1

is in compliance with the compressed breathing air specification as described by

NFPA 1989-2003

for a sample described as from the compressed gas source

Eagle Compressor, S/N 53526101

analyzed on __1/2/06__ as documented in Report No. _05-21963_.

DATE LAST SAMPLED: 12/20/2005
SAMPLING SCHEDULE: Quarterly
YOUR NEXT SAMPLE IS DUE:

3/20/2006

Richard A. Smith, C.I.H., Laboratory Director

Trace Analytics, Inc.
15768 Hamilton Pool Rd.
Austin, TX 78736  800-AIR-1024

LABORATORY ACCREDITED BY THE AMERICAN ASSOCIATION FOR LABORATORY ACCREDITATION IN THE CHEMICAL FIELD OF TESTING
RESULTS REPORTED RELATE ONLY TO THE ITEMS TESTED.
CERTIFICATE AND REPORT SHALL NOT BE REPRODUCED EXCEPT IN FULL WITHOUT THE WRITTEN PERMISSION OF THIS LABORATORY.
Appendix E – St. Paul Traveler’s Lab Results
Laboratory Work Order Number: 2006030712

Report Issued To: Joe Martin
Shipman's Fire Equipment
172 Cross Rd.
Waterford, CT 06385

Date Samples Received: 3/27/2006
Report Date: 3/28/2006

Location Sampled: Shipman's Fire Equipment
Sample Submitter: Joe Martin

<table>
<thead>
<tr>
<th>Sample ID</th>
<th>Sample Description</th>
<th>Results</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>mg/mL</td>
<td>µg</td>
</tr>
<tr>
<td>1</td>
<td>Providence Air #1</td>
<td>LT 0.11</td>
</tr>
<tr>
<td>2</td>
<td>Providence House Comp.</td>
<td>LT 0.085</td>
</tr>
<tr>
<td>3</td>
<td>Cranston F. D. House</td>
<td>LT 0.080</td>
</tr>
<tr>
<td>4</td>
<td>Providence Scott Cylinder</td>
<td>LT 0.075</td>
</tr>
<tr>
<td>Blank</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Results are based on an impinger volume of 10 milliliters.

<table>
<thead>
<tr>
<th>Analyte</th>
<th>Media type</th>
<th>LOQ</th>
<th>Reference Method</th>
<th>Analysis Date</th>
</tr>
</thead>
<tbody>
<tr>
<td>Hydrogen Cyanide</td>
<td>NaOH Imp</td>
<td>1.0 µg</td>
<td>Ion Chromatography - OSHA ID 120</td>
<td>3/27/2006</td>
</tr>
</tbody>
</table>

Please Note: The limits of quantitation (LOQs) listed are for normally processed samples. Sample requiring special processing (i.e. dilutions) may have elevated LOQs. N.A. = Not Applicable

WORKORDER COMMENTS:

The reported data relate only to the samples as received by the Laboratory. The reported air concentrations have been calculated using information supplied by the customer and have NOT been adjusted to represent a Time Weighted Average (TWA). "LT" indicates less than the limit of quantitation (LOQ). The contaminant may or may not be present at levels below this concentration. This report shall not be reproduced except in full, without written approval of the laboratory. The samples have not been blank corrected unless otherwise noted. Unless otherwise noted, all samples were received in satisfactory condition.

Approved by:

Tom Surveski  
QA Group Leader

Josef Chrzanowski  
Production Group Leader

George E. Johnson  
Group Leader

Marcel F. Baril  
Laboratory Director
Appendix F – Rhode Island Department of Health Water Quality Lab Report
### Bromochloromethane (ppb)
- Result: <1.0
- Standard: 1400.0h

### Dichlorodifluromethane (ppb)
- Result: <0.5
- Standard: 70m

### Hexachlorobutadiene (ppb)
- Result: <1.0
- Standard: 20.000h

### P-Isooctyltoluene (ppb)
- Result: <0.5
- Standard: 60m

### N-Propylbenzene (ppb)
- Result: <0.5
- Standard: 10m

### Tert-Butylnaphthalene (ppb)
- Result: <0.5
- Standard: 300m

### N-Butylbenzene (ppb)
- Result: <0.5
- Standard: 3400.0h

### Trichlorofluoromethane (ppb)
- Result: <0.5
- Standard: 1000h

### Isopropylbenzene (ppb)
- Result: <0.5
- Standard: 20.000h

### Naphthalene (ppb)
- Result: <0.5
- Standard: 1000h

### Decylbenzene (ppb)
- Result: <0.5
- Standard: 1000h

### 1,2,3-Trichlorobenzene (ppb)
- Result: <0.5
- Standard: 1000h

### 1,2,4-Trimethylbenzene (ppb)
- Result: <0.5
- Standard: 70m

### Methyl Tertiary Butyl Ether (ppb)
- Result: <1.0
- Standard: 40.000h

### Methyl Xylene (ppb)
- Result: <1.0
- Standard: 90m

### Trans-1,3-Dichloropropene (ppb)
- Result: <0.5
- Standard: 10m

### Cyanide (ppm)
- Result: <0.01
- Standard: 0.2m

### Fluoride (ppm)
- Result: <0.20
- Standard: 4m

---

June A. Swallow, Chief, Drinking Water Quality
(401) 222-6867

Feb 16, 2006

CANNON BUILDING, Three Capitol Hill, Providence, Rhode Island 02908-5097
Hearing/Speech Impaired, Dial 711 or Call 1-800-745-5555 (TTY)
Web Site: www.HEALTH.ri.gov
Appendix G – Rhode Island Analytical Lab report on FF Baker’s Turnout Gear wipe tests
CERTIFICATE OF ANALYSIS

Providence Fire Department
Attn: J. Curtis Varone
Deputy Assistant Chief
325 Washington Street
Providence, RI 02903

Date Received: 4/4/06
Date Reported: 4/12/06
P.O. #: 
Work Order #: 0604-05763

DESCRIPTION: Fire fighting apparel including: helmet, coat, pants, boots, gloves, and face mask.

The above items have been analyzed by our Warwick, R.I. laboratory with the attached results.


Occupational Safety and Heath Administration, Chemical Sampling, revised 7/15/2003, Cyanide Wipe Samples

Data qualifiers (if present) are explained in full at the end of a given sample’s analytical results.

Certification #: RI-033, MA-R1015, CT-PH-0508, ME-R1015, NH-253700 A & B,
USDA S-41844, NY-11726

If you have any questions regarding this work, or if we can be of further assistance, please contact us at (401) 737-8500.

Approved By:

[Signature]
Data Reporting
The following items were exposed to vapors, generated during a building fire. The following results are from wipe samples collected from each of the items.

<table>
<thead>
<tr>
<th>ITEM</th>
<th>Wipe Sample Area</th>
<th>Cyanide Detected</th>
<th>Units</th>
<th>Date Analyzed</th>
</tr>
</thead>
<tbody>
<tr>
<td>Helmet</td>
<td>Entire outer surface</td>
<td>14</td>
<td>ug</td>
<td>4/5/06</td>
</tr>
<tr>
<td>Coat</td>
<td>4 ft² (back)</td>
<td>&lt;0.5</td>
<td>ug/ft²</td>
<td>4/5/06</td>
</tr>
<tr>
<td>Pants</td>
<td>1 ft² (left pant leg)</td>
<td>&lt;2.0</td>
<td>ug/ft²</td>
<td>4/5/06</td>
</tr>
<tr>
<td>Glove</td>
<td>Entire outer surface (left hand)</td>
<td>2.2</td>
<td>ug</td>
<td>4/5/06</td>
</tr>
<tr>
<td>Mask</td>
<td>Entire outer surface</td>
<td>&lt;0.5</td>
<td>ug</td>
<td>4/5/06</td>
</tr>
<tr>
<td>Boot</td>
<td>Entire outer surface (left boot)</td>
<td>0.5</td>
<td>ug</td>
<td>4/5/06</td>
</tr>
</tbody>
</table>

Note: 1 ug is equal to one millionth of a gram.
Analytical Laboratories, Inc.  
QA/QC Report

Client: Providence Fire Department  
W.O. #: 0604-05763  
Date: 04/11/06

-Method Blank Results-

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Units</th>
<th>Results</th>
<th>Date Analyzed</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cyanide</td>
<td>mg/l</td>
<td>&lt;0.01</td>
<td>04/05/2006</td>
</tr>
</tbody>
</table>

-Laboratory Control Standard Results-

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Units</th>
<th>LCS Conc.</th>
<th>Detected Conc.</th>
<th>% Rec.</th>
<th>Date Analyzed</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cyanide</td>
<td>mg/l</td>
<td>0.10</td>
<td>0.097</td>
<td>97</td>
<td>04/05/2006</td>
</tr>
</tbody>
</table>

-Replicate Sample Results-

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Units</th>
<th>Sample #</th>
<th>Rep 1 Conc.</th>
<th>Rep 2 Conc.</th>
<th>Mean Conc.</th>
<th>Reported Value</th>
<th>RPD</th>
<th>Date Analyzed</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cyanide</td>
<td>mg/l</td>
<td>05450-5</td>
<td>&lt;0.01</td>
<td>&lt;0.01</td>
<td>&lt;0.01</td>
<td>0</td>
<td>0</td>
<td>04/05/2006</td>
</tr>
</tbody>
</table>

-Matrix Spike Results-

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Units</th>
<th>Sample #</th>
<th>Sample Conc.</th>
<th>Spike Conc.</th>
<th>Detected Conc.</th>
<th>% Rec.</th>
<th>Date Analyzed</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cyanide</td>
<td>mg/l</td>
<td>05301-3</td>
<td>&lt;0.01</td>
<td>0.10</td>
<td>0.098</td>
<td>98</td>
<td>04/05/2006</td>
</tr>
</tbody>
</table>
Test Methods for Evaluating Solid Waste

Volume IA: Laboratory Manual Physical/Chemical Methods
TEST METHODS FOR EVALUATING SOLID WASTE, PHYSICAL/CHEMICAL METHODS, SW-846, 3RD EDITION,

FINAL UPDATE 1
METHOD 9010A

TOTAL AND AMENABLE CYANIDE

1.0 SCOPE AND APPLICATION

1.1 Method 9010 is used to determine the concentration of inorganic cyanide (CAS Registry Number 57-12-5) in wastes or leachate. The method detects inorganic cyanides that are present as either soluble salts or complexes. It is used to determine values for both total cyanide and cyanide amenable to chlorination. The "reactive" cyanide content of a waste, that is, the cyanide content that could generate toxic fumes when exposed to mild acidic conditions, is not distilled by Method 9010 (refer to Chapter Seven). However, Method 9010 is used to quantify the concentration of cyanide from the reactivity test.

1.2 The titration procedure using silver nitrate with p-dimethylamino-benzal-rhodanine indicator is used for measuring concentrations of cyanide exceeding 0.1 mg/L (0.025 mg/250 mL of absorbing liquid).

1.3 The colorimetric procedure is used for concentrations below 1 mg/L of cyanide and is sensitive to about 0.02 mg/L.

1.4 This method was designed to address the problem of "trace" analyses (<1000 ppm). The method may also be used for "minor" (1000 ppm - 10,000 ppm) and "major" (>10,000 ppm) analyses by adapting the sample preparation techniques or cell path length. However, the amount of sodium hydroxide in the standards and the sample analyzed must be the same.

2.0 SUMMARY OF METHOD

2.1 The cyanide, as hydrocyanic acid (HCN), is released from samples containing cyanide by means of a reflux-distillation operation under acidic conditions and absorbed in a scrubber containing sodium hydroxide solution. The cyanide in the absorbing solution is then determined colorimetrically or titrmetrically.

2.2 In the colorimetric measurement, the cyanide is converted to cyanogen chloride (CNCl) by reaction of cyanide with chloramine-T at a pH less than 8. After the reaction is complete, color is formed on the addition of pyridine-barbituric acid reagent. The absorbance is read at 578 nm for the complex formed with pyridine-barbituric acid reagent and CNCl. To obtain colors of comparable intensity, it is essential to have the same salt content in both the sample and the standards.

2.3 The titration measurement uses a standard solution of silver nitrate to titrate cyanide in the presence of a silver sensitive indicator.

3.0 INTERFERENCES

3.1 Interferences are eliminated or reduced by using the distillation procedure. Chlorine and sulfide are interferences in Method 9010.
5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Reagent water. All references to water in this method refer to reagent water, as defined in Chapter One.

5.3 Reagents for sample collection, preservation, and handling

5.3.1 Sodium arsenite (0.1N), NaAsO₂. Dissolve 3.2 g NaAsO₂ in 250 mL water.

5.3.2 Ascorbic acid, C₆H₇O₆.

5.3.3 Sodium hydroxide solution (50%), NaOH. Commercially available.

5.3.4 Acetic acid (1.6M) CH₃COOH. Dilute one part of concentrated acetic acid with 9 parts of water.

5.3.5 2,2,4-Trimethylpentane, C₁₃H₂₆.

5.3.6 Hexane, C₆H₁₄.

5.3.7 Chloroform, CHCl₃.

5.4 Reagents for cyanides amenable to chlorination

5.4.1 Calcium hypochlorite solution (0.35M), Ca(OCl)₂. Combine 5 g of calcium hypochlorite and 100 mL of water. Shake before using.

5.4.2 Sodium hydroxide solution (1.25N), NaOH. Dissolve 50 g of NaOH in 1 liter of water.

5.4.3 Sodium arsenite (0.1N). See Step 5.3.1.

5.4.4 Potassium iodide starch paper.

5.5 Reagents for distillation

5.5.1 Sodium hydroxide (1.25N). See Step 5.4.2.

5.5.2 Bismuth nitrate (0.062M), Bi(NO)₃ • 5H₂O. Dissolve 30 g Bi(NO)₃ • 5H₂O in 100 mL of water. While stirring, add 250 mL of glacial acetic acid, CH₃COOH. Stir until dissolved and dilute to 1 liter with water.

5.5.3 Sulfamic acid (0.4N), H₂NSO₃H. Dissolve 40 g H₂NSO₃H in 1 liter of water.

9010A - 3

Revision 1
July 1992

6.0 SAMPLE COLLECTION, PRESERVATION AND HANDLING

6.1 All samples must be collected using a sampling plan that addresses the considerations discussed in Chapter Nine.

6.2 Samples should be collected in plastic or glass containers. All containers must be thoroughly cleaned and rinsed.

6.3 Oxidizing agents such as chlorine decompose most cyanides. To determine whether oxidizing agents are present, test a drop of the sample with potassium iodide-starch test paper. A blue color indicates the need for treatment. Add 0.1N sodium arsenite solution a few mL at a time until a drop of sample produces no color on the indicator paper. Add an additional 5 mL of sodium arsenite solution for each liter of sample. Ascorbic acid can be used as an alternative although it is not as effective as arsenite. Add a few crystals of ascorbic acid at a time until a drop of sample produces no color on the indicator paper. Then add an additional 0.6 g of ascorbic acid for each liter of sample volume.

6.4 Aqueous samples must be preserved by adding 50% sodium hydroxide until the pH is greater than or equal to 12 at the time of collection.

6.5 Samples should be chilled to 4°C.

6.6 When properly preserved, cyanide samples can be stored for up to 14 days prior to sample preparation steps.

6.7 Solid and oily wastes may be extracted prior to analysis by method 9013. It uses a dilute NaOH solution (pH = 12) as the extractant. This yields extractable cyanide.

6.8 If fatty acids, detergents, and surfactants are a problem, they may be extracted using the following procedure. Acidify the sample with acetic acid (1.6M) to pH 6.0 to 7.0.

CAUTION: This procedure can produce lethal HCN gas.

Extract with isooctane, hexane, or chloroform (preference in order named) with solvent volume equal to 20% of the sample volume. One extraction is usually adequate to reduce the compounds below the interference level. Avoid multiple extractions or a long contact time at low pH in order to keep the loss of HCN at a minimum. When the extraction is completed, immediately raise the pH of the sample to above 12 with 50% NaOH solution.
7.2.4 If samples are known or suspected to contain nitrate or nitrite, or if bismuth nitrate was added to the sample, add 50 mL of 0.4N sulfamic acid solution through the air inlet tube. Mix for three minutes.

Note: Excessive use of sulfamic acid could create method bias.

7.2.5 Slowly add 50 mL of 18M sulfuric acid through the air inlet tube. Rinse the tube with water and allow the airflow to mix the flask contents for three minutes. Add 20 mL of 2.5N magnesium chloride through the air inlet and wash the inlet tube with a stream of water.

7.2.6 Heat the solution to boiling. Reflux for one hour. Turn off heat and continue the airflow for at least 15 minutes. After cooling the boiling flask, and closing the vacuum source, disconnect the gas scrubber.

7.2.7 Transfer the solution from the scrubber into a 250-mL volumetric flask. Rinse the scrubber into the volumetric flask. Dilute to volume with water.

7.2.8 If the manual spectrophotometric determination will be performed, proceed to Step 7.3.1. If the titration procedure will be performed, proceed to Step 7.7.

7.3 Manual spectrophotometric determination

7.3.1 Pipet 50 mL of the scrubber solution into a 100-mL volumetric flask. If the sample is later found to be beyond the linear range of the colorimetric determination and redistillation of a smaller sample is not feasible, a smaller aliquot may be taken. If less than 50 mL is taken, dilute to 50 mL with 0.25N sodium hydroxide solution.

NOTE: Temperature of reagents and spiking solution can affect the response factor of the colorimetric determination. The reagents stored in the refrigerator should be warmed to ambient temperature before use. Samples should not be left in a warm instrument to develop color, but instead they should be aliquoted to a cuvette immediately prior to reading the absorbance.

7.3.2 Add 15 mL of 1M sodium phosphate solution and mix. Add 2 mL of chloramine-T and mix. Some distillates may contain compounds that have chlorine demand. One minute after the addition of chloramine-T, test for excess chlorine with KI-starch paper. If the test is negative, add 0.5 mL chloramine-T. After one minute recheck with KI-starch paper. Continue to add chloramine-T in 0.5 mL increments until an excess is maintained. After 1 to 2 minutes, add 5 mL of pyridine-barbituric acid solution and mix.

7.3.3 Dilute to 100 mL with water and mix again. Allow 8 minutes for color development and then read the absorbance at 578 nm in a 1-cm cell within 15 minutes. The sodium hydroxide concentration will be 0.125N.

9010A - 7
Revision 1
July 1992
where:

\[ \begin{align*}
A &= \mu g/L \text{ CN}^- \text{ read from standard curve.} \\
B &= \text{mL of sample after preparation of colorimetric analysis (100 mL recommended).} \\
C &= \text{mL of sample after distillation (250 mL recommended).} \\
D &= \text{mL of original sample for distillation (500 mL recommended).} \\
E &= \text{mL used for colorimetric analysis (50 mL recommended).}
\end{align*} \]

7.7 Titration Procedure

7.7.1 Transfer the gas scrubber solution or a suitable aliquot from the 250-mL volumetric flask to a 500-mL Erlenmeyer flask. Add 10-12 drops of the rhodanine indicator.

7.7.2 Titrate with standard 0.0192N silver nitrate to the first change in color from yellow to brownish-pink. The titration must be performed slowly with constant stirring. Titrate a water blank using the same amount of sodium hydroxide and indicator as in the sample. The analyst should be familiar with the endpoint of the titration and the amount of indicator to be used before actually titrating the samples. A 5-mL buret may be conveniently used to obtain a precise titration.

NOTE: The titration is based on the following reaction:

\[ \text{Ag}^+ + 2\text{CN}^- \rightarrow [\text{Ag(CN)}_2]^-. \]

When all of the cyanide has complexed and more silver nitrate is added, the excess silver combines with the rhodanine indicator to turn the solution yellow and then brownish-pink.

7.7.3 Calculation - If the titrimetric procedure is used, calculate concentration of CN\(^-\) in \( \mu g/L \) in the original sample as follows:

\[ \text{CN}^- (\mu g/L) = \frac{(A - B)}{C} \times D \times \frac{F}{F} \times \frac{2 \text{ mole CN}^-}{1 \text{ eq. AgNO}_3} \times \frac{26.02 \text{ g CN}^-}{1 \text{ mole CN}^-} \times \frac{1 \times 10^6 \mu g}{1 g} \]

where:

\[ \begin{align*}
A &= \text{mL of AgNO}_3 \text{ for titration of sample.} \\
B &= \text{mL of AgNO}_3 \text{ for titration of blank.} \\
C &= \text{mL of sample titrated (250 recommended).} \\
D &= \text{actual normality of AgNO}_3 (0.0192N \text{ recommended).} \\
E &= \text{mL of sample after distillation (250 recommended).} \\
F &= \text{mL of original sample before distillation (500 recommended).}
\end{align*} \]
10.0 REFERENCES


7. Elly, C.T. J. Water Pollution Control Federation 1968, 40, 848-856.


FIGURE 2.
APPARATUS FOR CYANIDE DISTILLATION

- Allihn Condenser
- Air Inlet Tube
- Connecting Tubing
- Gas Scrubber
- One-Liter Boiling Flask
- Suction
METHOD 9010A
(Continued)

7.4.1 Prepare a series of cyanide standards through dilution

7.4.2 Perform colorimetric analysis of standards

7.4.3 Distill at least two standards to check distillation recovery

7.4.4 Prepare standard curve of absorbances

7.4.5 Check efficiency of sample distillation

7.5.1 Distill standards in same manner as samples

7.5.2 Prepare standard curve of absorbances

7.7.2 Titrate sample to flask; add rhodamine indicator

7.7.3 Calculate concentration of cyanide in sample

STOP

STOP
Appendix H – Tests on FF Baker’s SCBA and mask
FUNCTIONAL TESTING WORKSHEET FOR SCOTT AIR-PAK®
E-Z FLO® REGULATOR (2216 & 4500 psi)

SERVICE CENTER NAME: E-6
DATE: 5/30/05

ADDRESS: ____________________________
No. and Street/P.O. Box State Or Province Country Zip or Postal Code
TECHNICIAN'S NAME: ____________________________
PHONE NUMBER: ____________________________

REGULATOR OWNER: ____________________________

ADDRESS: ____________________________
No. and Street/P.O. Box City State Or Province Country Zip or Postal Code
CONTACT PERSON: ____________________________
PHONE NUMBER: ____________________________

REGULATOR PART NUMBER: 803572-01 SERIAL NUMBER: A+00343/A+00112

REGULATOR SET-UP AND FUNCTIONAL TEST

Manual Shut-Off Functional Test (For donning switch and E-Z Flo regulators)
• Was breathing normal with manual shut-off deactivated? (Must be free and unrestricted):
• Did all flow stop with manual shut-off activated? (No flow allowed):
• Did manual shut-off operate properly? (Must move smoothly when depressed and return fully when released):
• Manual shut-off reset pressure (must be between -2.5 and -5.0 in. H₂O for donning switch regulators and -3.0 and -5.0 in. H₂O for E-Z Flo regulators):

Static Pressure Test
• Chamber pressure indication (must be between +0.8 and +1.5 in. H₂O):

Regulator Flow Test
• Was the regulator able to produce the required flow at 65 psig inlet pressure? (minimum flow 400 lpm):

Purge Flow Test
• Purge flow indicator halfway between the 125 and 225 indication on gauge 127 (Purge knob fully open):
• Did all flow stop with purge knob turned fully "OFF"? (No flow allowed):

Alarm Test
• Did Vibrialert alarm actuate at 135 psig inlet pressure? (Vibrialert alarm shall actuate):
• Did (Optional) Beacon Alarm alarm actuate at 145 psig inlet pressure? (Beacon Alarm shall actuate):
• Did Vibrialert and (Optional) Beacon Alarms continue to actuate at 160 psig inlet pressure? (Vibrialert and (Optional) Beacon Alarms shall continue to actuate):
• Did Vibrialert and (Optional) Beacon Alarms stop actuating at 110 psig inlet pressure? (Vibrialert and (Optional) Beacon Alarms shall stop actuating):

External Leakage Test
• Did leakage occur at 100 psig inlet pressure without cap assembly installed? (pressure must remain above 00 psig for 30 seconds):
• Did leakage occur at 100 psig inlet pressure with cap assembly installed? (pressure must remain above 00 psig for 30 seconds):

Breathing Test
• Was breathing normal at 85-110 psig inlet pressure? (must be free and unrestricted):
• Did breathing remain normal at 145-165 psig inlet pressure? (must remain free and unrestricted):
• Did alarm actuate at 145-165 psig inlet pressure? (Alarm shall actuate; beacon shall be visible):
• Did alarm stop actuating at 85-110 psig inlet pressure? (Alarm shall not actuate; beacon shall not be visible):
• Did a free flow of air occur with purge knob turned fully "ON"? (A free flow of air shall occur):
• Did the free flow of air stop with purge knob turned fully "OFF"? (No flow allowed):

NOTE: This form is intended to be used in conjunction with "Air-Pak Overhaul Manual, H/S 5445."

SCOTT HEALTH & SAFETY
309 W. Crowell Ave., Monroe, NC 28112 • Tel.: 704-282-8400 • Fax: 704-282-8423
E-mail: hssales@scottaviation.com Website: www.scottaviation.com
H/S 5697E 5/00
Printed in U:
SCOTT PosiChek3
Visual / Functional Test Results
PFD

PosiChek3 PM
calibration was up
to date when this
test was
performed.

Apparatus Tested
Location: AIR SUPPLY ROOM
Other ID:

Auxiliary IDs
Facepiece / Head Har AV-2000(CS) SCOTT
Regulator 19700343
Reducer 19700110
Low Pressure Alarm VIBRALERT
Cylinder 30/SCI/CARBON
Airline Attachment NO
Harness WIRE/KEVLAR

Visual Inspection
Facepiece / Head Harness Pass
Backframe/Harness Pass
Cylinder Pass
Alarms Pass
Hoses Pass

Functional Tests
Facepiece Leak Test Pass 0.1 in. H2O
Exhalation Pressure Pass 1.9 in. H2O
Remote Pressure Gauge Pass

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<thead>
<tr>
<th>Pressure</th>
<th>1000 PSI</th>
<th>2000 PSI</th>
<th>3000 PSI</th>
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<tr>
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<td>Pass</td>
<td>1945</td>
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<td>2844 PSI</td>
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Alarm(s) Activation Pass 1146 PSI
Air Saver Switch Pass -4.0 in. H2O
Static Facepiece Pressure Pass 0.9 in. H2O
Primary Reducer Lockup Pass 89 PSI
Primary Creep Pass -7 PSI
Low Cylinder Transfer Pr Pass 1146 PSI
Secondary Reducer Lockup Pass 158 PSI
Secondary Reducer Creep Pass -5 PSI
Purge Flow Test Pass 173 L/min
High Pressure Leakage Pass 22 PSI
Secondary Pr. at High Cyl. Pass 146 PSI

Scott Air-Pak 4.5 - E-6 4/27/2006 12:52:32 PM
Standard Work Rate

Scott Air-Pak 4.5 - E-6 4/27/2006 12:52:32 PM
Maximum Work Rate (102 Liter Minute Vol)

Minimum Maximum Breathing Results Minimum Maximum
0.6 in. H2O 2.0 in. H2O Pass Facepiece Pressure 0.3 in. H2O 2.8 in. H2O Pass

4/27/2006 2:01:04 PM ALAN R MOFFAT: Sanitized regulator, facepiece provided with SCBA was used in test.

Tested by: ALAN R MOFFAT
Service Center: AIR SUPPLY/PROVIDENCE FIRE EP

Signature

Page 1 of 1 Version 3.22
# Replacement Parts Log:

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<tr>
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<tr>
<td>1000 5449</td>
<td>Conical spring</td>
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<tr>
<td>1080 4+63</td>
<td>Retaining ring</td>
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<tr>
<td>803652-01</td>
<td>Diaphragm + Valve assembly</td>
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<tr>
<td>1000 6455</td>
<td>O-ring on piston</td>
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**Comments:**

Regulator Cover dented, diaphragm torn, retaining ring cracked.
SCOTT HEALTH & SAFETY
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FUNCTIONAL TESTING WORKSHEET FOR SCOTT AIR-PAK® 2.2/3.0/4.5 PRESSURE REDUCER

SERVICE CENTER NAME: X E-6
DATE: 05 Oct 05

ADDRESS:

TECHNICIAN'S NAME: M ROFF E-ALAN
PHONE NUMBER:

PRESSURE REDUCER OWNER:

ADDRESS:

CONTACT PERSON:

PHONE NUMBER:

PRESSURE REDUCER PART NUMBER: 802220-02
TYPE OF REDUCER: 2.2 3.0 4.5
SERIAL NUMBER: 19700110/19700343

PRESSURE REDUCER SET-UP AND FUNCTIONAL TEST

Primary and Secondary Set-Up and Adjustment
• Secondary pressure at 200 psi inlet pressure (should be between 145-165 psi with no increase above 165 psi within 30 seconds after lock-up):
• Secondary pressure at 400 psi (for 2.2), 550 psi (for 3.0) or 900 psi (for 4.5) inlet pressure (Should be between 145 and 165 psi with no increase above 165 psi within 30 seconds after lock-up):
• Primary pressure at 900 psi (for 2.2), 950 psi (for 3.0) or 1500 psi (for 4.5) inlet pressure (Should be between 85 and 110 psi with no increase above 110 psi within 30 seconds after lock-up):

Primary Pressure at High Cylinder Pressure
• Primary pressure at 2216 psi (for 2.2), 3000 psi (for 3.0) or 4500 psi (for 4.5) inlet pressure (must be between 85 and 110 psi with no increase above 110 psi within 30 seconds after lock-up):

Automatic Transfer and Secondary Pressure at High Cylinder Pressure
• Did the automatic transfer occur? (Automatic transfer shall occur):
• Secondary pressure after transfer at 2216 psi (for 2.2), 3000 psi (for 3.0) or 4500 psi (for 4.5) inlet pressure (Must be between 140 and 165 psi with no increase above 165 psi within 30 seconds after lock-up):
• Primary pressure after return from transfer (must be between 85 and 110 psi with no increase above 110 psi within 30 seconds after lock-up):

External Leakage
• Was external leakage present? (No leakage allowed):

If yes, indicate the location with a check-mark in the appropriate box below:
- High Pressure Hose/Inlet Seal
- Top Cover
- Top Cover/Body Seal
- Seat Retainer Seat(s)
- Relief Valve
- Gaugeline Block/Body Seal
- Low Pressure Hose/Outlet Seal
- Outlet Manifold/Body Seal

Primary Lock-Up at Low Cylinder Pressure
• Primary pressure at 800 psi (for 2.2), 950 psi (for 3.0) or 1500 psi (for 4.5) inlet pressure (Must be between 85 and 110 psi with no increase above 110 psi within 30 seconds after lock-up):

Primary Flow Test
• Did primary produce the required flow at 900 psi (for 2.2), 950 psi (for 3.0) or 1500 psi (for 4.5) inlet pressure (minimum flow 400 lpm):
• Primary pressure during flow test (must remain above 40 psi):

Low Cylinder Transfer Pressure
• Did the low cylinder transfer occur? (Low cylinder transfer shall occur):
• Inlet pressure when transfer occurs (must be between 510 and 600 psi (for 2.2), 690 and 810 psi (for 3.0) or 1000 and 1250 psi (for 4.5)):
• Secondary pressure after transfer (must be above 135 psi):

Secondary Alarm Test (Optional with Bell Alarm)
• Did alarm activate? (secondary alarm must activate):
• Inlet pressure when alarm activates (must be between 510 and 600 psi (for 2.2), 690 and 810 psi (for 3.0), 1000 and 1250 psi (for 4.5)):

Secondary Pressure at Low Cylinder Pressure
• Secondary pressure during 4 lpm flow test at 400 psi (for 2.2), 550 psi (for 3.0) or 900 psi (for 4.5) inlet pressure (must be between 140 and 160 psi):

Secondary Flow Test
• Secondary pressure during 25 lpm flow test at 400 psi (for 2.2), 550 psi (for 3.0) or 900 psi (for 4.5) inlet pressure (must be between 135 and 160 psi):
• Did secondary produce the required flow at 400 psi (for 2.2), 550 psi (for 3.0) or 900 psi (for 4.5) inlet pressure? (Minimum flow 400 lpm):
• Secondary pressure during flow test (must remain above 105 psi):
• Secondary pressure during 25 lpm flow test at 300 psi inlet pressure (must be between 135 and 160 psi):

NOTE: This form is intended to be used in conjunction with "Air-Pak 2.2/3.0/4.5 Overhaul Manual, H/S 5445."
## REPLACEMENT PARTS LOG:

<table>
<thead>
<tr>
<th>PART NUMBER</th>
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<td>804085-01</td>
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Comments:

Steps frayed
Appendix I - Rhode Island Hospital Lab Procedures for whole blood cyanide testing
CYANIDE—WHOLE BLOOD

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METHOD: Micro-Diffusion and Spectroscopy

PRINCIPLE:

Any analytical method purported to support the emergency diagnosis and treatment of cyanide poisoning must provide results on a timely basis because this toxicant acts rapidly. The laboratory can provide analytical results within a time that will allow effective therapy with specific antidotes; contrary to common belief, survival for several hours after ingestions of even supralethal amounts of cyanide, particularly with supportive treatment, is not uncommon.

Cyanide in whole blood is relatively stable for several days, even at ambient temperature, because of the tight binding of cyanhemoglobin. Cyanide in plasma is rapidly converted to thiocyanate. Hence, whereas whole blood or gastric specimens may be analyzed for cyanide, on should analyze plasma, serum, and urine specimens for thiocyanate because their cyanide content may at most be only moderately increased, even in acute poisoning.

In this procedure cyanogen bromide reacts with pyridine/p-phenylenediamine to produce a colored complex. Both thiocyanate and cyanide will undergo the following reactions:

\[
\text{KSCN} + 4 \text{Br}_2 + 4 \text{H}_2\text{O} \rightarrow \text{CNBr} + 6 \text{HBr} + \text{H}_2\text{SO}_4 + \text{KBr}
\]

\[
\text{HCN} + \text{Br}_2 \rightarrow \text{CNBr} + \text{HBr}
\]

\[
\text{CNBr} + \text{pyridine/p-phenylenediamine} \rightarrow \text{pyridine dye}
\]

SAMPLE COLLECTION

1. Whole blood containing EDTA as an anticoagulant.
2. No special storage required.

REAGENTS

2. Arsenic Trioxide Solution, 0.1 mol/l, pH 7.6. Dissolve 2.0 g arsenic trioxide in 100 ml of 0.1 mol/l sodium hydroxide. Heat solution briefly, cool, and adjust pH to 7.6 with concentrated hydrochloric acid.
3. Phenylethylenediamine, 0.2 %. To 50 ml 0.5 N hydrochloric acid, add 100 mg phenylethylenediamine.
4. Pyridine Reagent. Mix 30 ml of pyridine, 5 ml of concentrated hydrochloric acid, and 20 ml distilled water.
6. Hydrochloric Acid, 1 N. To 400 ml distilled water, add 41.7 ml concentrated hydrochloric acid and dilute to 500 ml with distilled water.
7. Sodium Hydroxide, 0.1 N. Into a liter mixing cylinder containing 900 ml distilled water, add 4 g sodium hydroxide and dilute to 1000 ml with distilled water.
8. Sulfuric Acid, 10 Molar. To 40 ml distilled water, carefully add 55.6 ml concentrated sulfuric acid and mix carefully. When cooled to ambient temperature, dilute to 100 ml with distilled water.

STANDARDS

1. Stock Cyanide Standard, 1 mg/ml. To 80 ml 0.1 N sodium hydroxide, add 250 mg potassium cyanide and dilute to 100 ml with the 0.1 N sodium hydroxide.
2. Sub-Stock Cyanide Standard, 0.01 mg/ml. Into a 100 ml volumetric flask, add 1 ml stock cyanide standard and dilute to 100 ml with distilled water.
3. Working Cyanide Standards. To 2 ml cyanide free whole blood, add 100 and 200 ul sub-stock cyanide standard. These correspond to 50 and 100 ug/dl, respectively.

QUALITY CONTROL

2. Refer to the Toxicology Quality Control Manual for control tolerances.

INSTRUMENTAL PARAMETERS

1. Wavelength — 490 nm
2. Heat Source, 40–50 °C

PROCEDURE

1. Label Conway diffusion cells as working standards, control, and unknown samples.
2. Place a layer of vacuum grease along the entire lip of cell cover.
3. Pipet 0.5 ml of 0.1 N sodium hydroxide into the center well of each diffusion cell.
4. Pipet 2.0 ml of the working standards, control, and unknown samples into the outer ring of the appropriate diffusion cells.
5. Add 0.5 ml 10 Molar sulfuric acid to the outer ring of each diffusion cell, and position the cell cover without delay.
6. Briefly tilt and rotate the diffusion cells to mix the sulfuric acid with the whole blood, being careful that none of the samples spills over into the center well.
7. Position the diffusion cells in a uniformly heated environment of 40–50°C and incubate for ten minutes.
8. Remove the diffusion cells to ambient temperature and remove the cell covers.
9. Into 10 X 75 mm test tubes, add 0.1 ml of the sodium hydroxide from the center well. Prepare a blank with 0.1 N sodium hydroxide.
10. Add 0.5 ml 1 N hydrochloric acid and mix.
11. Add 50 ul of saturated bromine water and mix.
12. Add 200 ul arsenic trioxide and mix.
13. Add 0.8 ml of chromogenic reagent and mix. Let stand for at least three minutes.
14. Measure absorbances on a spectrophotometer at 490 nm within twelve minutes.

CALCULATIONS

Construct a standard curve on linear graph paper by plotting the absorbances of the working standards versus the concentrations. The plot should yield a straight line. Determine the concentration of the unknown from this standard curve.
CALCULATIONS

Construct a standard curve on linear graph paper by plotting the absorbances of the working standards versus the concentrations. The plot should yield a straight line. Determine the concentration of the unknown from this standard curve.

LIMITATION OF PROCEDURE

1. Minimum Detectable Level — 1 ug/dl
2. Interferences — no interferences have been identified.
3. Linearity - up to 400 ug/dl.

REPORTING RESULTS

1. Normal Values — cyanide is normally present in the blood of healthy individuals at concentrations up to 20 ug/dl, the result of vitamin B 12 metabolism and of environmental factors such as cigarette smoking and ingestion of plants and plant products containing cyanide glucoside, and amygdalin.
2. Toxicity — anything greater than 20 ug/dl will show some signs of mild toxicity.
3. For Critical Results, refer to "Critical Values" section of the procedure manual.

REFERENCE


AUTHOR: William C. Bastan, Ph.D.