Montreal Protocol
on Substances that Deplete the Ozone Layer

Report of the
Halons Technical Options Committee
December 2018

Technical Note #4

RECOMMENDED PRACTICES FOR RECYCLING
HALON AND OTHER HALOGENATED GASEOUS FIRE EXTINGUISHING AGENTS

The text of this report is composed in Times New Roman

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Preface

Technical Note #4, *Recommended Practices for Recycling Halons and Other Halogenated Gaseous Fire Extinguishing Agents*, replaces the chapters on this subject that have been part of previous Assessment Reports of the UNEP Halon Technical Options Committee (HTOC). Reports beginning with the 2014 Assessment Report contain an abbreviated chapter that briefly introduces the subject of *Recycling Halon and Other Gaseous Halogenated Fire Extinguishing Agents* and refers the interested reader to this document. The HTOC elected to take this approach as much of the information that, while important to understand when developing strategies for recycling halons and alternatives, has been largely reported in prior editions of the Assessment Reports, which contained few changes or updates. As such, it was deemed by the HTOC to make the Recycling subject a stand-alone document that is referenced by future Assessment Reports. By this approach those having particular interest in the technical aspects of the Recycling subject can access a self-contained document. One final note – the emission reduction strategies apply equally well to the halocarbon alternatives to halon and the HTOC encourages the readers of both the Assessment Reports and this document to integrate these strategies into their operations, policies, and guidelines when employing all halogenated gaseous fire extinguishing agents. This includes halons, hydrochlorofluorocarbons (HCFCs), hydrofluorocarbons (HFCs), perfluorocarbons (PFCs), fluoroketones (FKs), and a hydrobromofluoro-olefin (alkene), that have been or are now used for fire-fighting applications. Most recently a hydrochlorofluoro-olefin and a fluoroiodocarbon (FIC) are being tested for potential use in fire-fighting applications.
1.0 Introduction

Prior to the halt in production of halons, replenishment agent to recharge extinguishers and extinguishing systems had a fairly simple supply chain from manufacturer to servicing company to the end user. With such a short supply chain, the quality assurance needs of all organisations were readily achieved; or, in the rare case of Out of Specification (OOS) agent, problems were easily traced back to the source and corrective action taken.

Today, we no longer have newly manufactured halons and the fire protection industry must rely on “used” halons for the recharge of extinguishers and extinguishing systems. In the case of other halogenated gaseous fire extinguishing agents, while they are still being manufactured, some of the replenishment agent used for the recharge of extinguishers and extinguishing systems is from systems or extinguishers removed from service. That being the case, the industry faces the same challenges in ensuring the quality, especially purity, for both halons and other halogenated gaseous fire extinguishing agents used for replenishment. The source of replenishment agent has thus shifted from a handful of agent manufacturers around the world to literally millions of end users who own extinguishers or extinguishing systems and who may at some point offer the agent up for recycling / reclamation. Furthermore, the condition of the agent at its entry (or re-entry) point into the market has shifted from newly manufactured agents with an extremely high level of purity to “used” agent that can have any of several types of impurities.
2.0 Standard Methods of Treatment of Halons and Other Gaseous Halogenated Fire Extinguishing Agents

In the fire protection industry there are several terms used to describe the treatments of halons and other halogenated gaseous fire extinguishing agents to prepare them for possible redeployment:

- **Reuse**: To remove agent cylinder or extinguisher from one application and re-install in another application.
- **Recover**: To remove agent in any condition from an extinguisher or extinguishing system cylinder and store it in an external container without necessarily testing or processing it in any way.
- **Recycle**: To clean recovered agent for reuse without meeting all of the requirements for reclamation. In general, recycled agent has its pressurizing nitrogen removed in addition to being processed to only reduce moisture and particulate matter.
- **Reclaim**: To reprocess agent to a purity specified in applicable standards and to use a certified laboratory to verify this purity using the analytical methodology as prescribed in those standards. Reclamation is the preferred method to achieve the highest level of purity. Reclamation requires specialized machinery usually not available at a servicing company.

It is common for the expression “recycle” to include both the “reclaim” and “recycle” treatments described above. However, it is essential for everyone in the supply chain to understand the difference and to employ the correct method.

In order to have a credible agent resupply industry, the “used” agents must be properly processed in order to remove impurities and return the agent to a purity level consistent with newly manufactured agent or other appropriate standard. Furthermore, the participants in the agent resupply industry must have the technical ability to test and certify that the agents being offered for replenishment are indeed free of impurities. Without that ability rigorously applied, there can be no credible fire extinguishing agent resupply industry.
### 3.0 Specifications for Agent Treatment, Testing and Certification

The most common halogenated gaseous fire extinguishing agents in use today, including a relatively new agent introduced into hand portables on civil aircraft are listed in Table 3.1 along with some of their related purity standards:

<table>
<thead>
<tr>
<th>Designation</th>
<th>Chemical name</th>
<th>Purity Standard(s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Halon 1301</td>
<td>Bromotrifluoromethane (CF$_3$Br)</td>
<td>ASTM D5632/D5632M-17 [1]</td>
</tr>
<tr>
<td></td>
<td></td>
<td>ISO 7201-1:1989 Fire protection -- Fire extinguishing media --</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Halogenated hydrocarbons -- Part 1: Specifications for halon 1211 and halon 1301</td>
</tr>
<tr>
<td>Halon 1211</td>
<td>Bromochlorodifluoromethane (CF$_2$BrCl)</td>
<td>ASTM D7673-10 [2]</td>
</tr>
<tr>
<td></td>
<td></td>
<td>ISO 7201-1:1989 Fire protection -- Fire extinguishing media --</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Halogenated hydrocarbons -- Part 1: Specifications for halon 1211 and halon 1301</td>
</tr>
<tr>
<td>Halon 2402</td>
<td>1,2-Dibromotetrafluoroethane (C$_2$Br$_2$F$_4$)</td>
<td>GOST 15899-93 [3]</td>
</tr>
<tr>
<td>HCFC Blend B</td>
<td>Blend comprised primarily of 2,2-dichloro-1,1,1-trifluoroethane,</td>
<td>ASTM D7122-17 [4]</td>
</tr>
<tr>
<td></td>
<td>(CF$_3$CCl$_2$H, Ar, and CF4)</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>[5]</td>
</tr>
<tr>
<td>HFC-227ea</td>
<td>1,1,1,2,3,3,3-Heptfluoropropane (CF$_3$CHFCF$_3$)</td>
<td>ASTM D6064-11(2015) [7]</td>
</tr>
<tr>
<td>HFC-236fa</td>
<td>1,1,1,3,3,3-hexafluoropropane (CF$_3$CH2CF$_3$)</td>
<td>ASTM D6541-11(2015) [8]</td>
</tr>
<tr>
<td>FK-5-1-12</td>
<td>Dodecafluoro-2-methylpentane-3-one</td>
<td>ISO 14520 – Part 5 [9]</td>
</tr>
<tr>
<td>HBFO-1233xf(B)</td>
<td>2-Bromo-3,3,3-Trifluoro-1-Propene (CF$_3$CBr=CH$_2$), (aka 2-BTP)</td>
<td>ASTM D8060-17 [10]</td>
</tr>
</tbody>
</table>
The requirements for quality of these agents are also contained in NFPA 2001 [11] and the International Standards Organisation (ISO) 14520 series of standards [9]. The typical requirements are summarized in Table 3.2.

**Table 0.1: Typical Requirements**

<table>
<thead>
<tr>
<th>Property</th>
<th>Halocarbons (1)</th>
<th>Halon 1211 (2)</th>
<th>Halon 1301 (2)</th>
<th>Halon 2402 (3)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Purity, % (mol/mol)</td>
<td>99.0 min</td>
<td>99.0 min</td>
<td>99.6 min</td>
<td>99.5 min</td>
</tr>
<tr>
<td>Acidity, ppm by mass</td>
<td>3.0 max</td>
<td>3.0 max</td>
<td>3.0 max</td>
<td></td>
</tr>
<tr>
<td>Water content, ppm by mass</td>
<td>10 max</td>
<td>20 max</td>
<td>10 max</td>
<td>30 max</td>
</tr>
<tr>
<td>Non-volatile residue, % (mol/mol)</td>
<td>0.05</td>
<td>0.01</td>
<td>0.01</td>
<td></td>
</tr>
<tr>
<td>Halogen Ion</td>
<td>Passes test</td>
<td>Passes test</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Suspended matter or sediment</td>
<td>None visible</td>
<td>None visible</td>
<td>None visible</td>
<td></td>
</tr>
<tr>
<td>Color</td>
<td>Report Value</td>
<td>Report Value</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Note 2: According to ISO 7201-1 [12]
Note 3: According to GOST 15899-9 [3]

The following are some suggested procedures for testing the requirements in Tables 3.1 and 3.2:

- **Purity**: Determine the purity by gas-liquid chromatography (GC), using generally accepted laboratory techniques. If this test indicates the presence of unidentified impurities, then determination by gas-liquid chromatography/mass spectrometry (GC/MS) is recommended.

- **Acidity**: Determine the acidity by the appropriate method specified in ISO 3363 [13] or Analytical Procedures for AHRI Standard 700 [14].

- **Water content**: Determine the water content by the orthodox Karl Fischer method [15], Analytical Procedures for AHRI Standard 700 [14], or by any other method giving equivalent results.

- **Non-volatile residue**: Determine the non-volatile residue by the method specified in ISO 5789 [16] or Analytical Procedures for AHRI Standard 700 [14].

- **Halogen ions**: Mix 5 g of the sample with 5 ml of absolute methanol containing several drops of a saturated methanolic silver nitrate (AgNO₃) solution. The resulting solution shall exhibit no turbidity or precipitation of silver halide.

- **Suspended matter or sediment**: Examine the liquid phase of the sample visually.

- **Color**: ASTM D2108-10 (2015) [17]
Slight differences in the parameter limits are inherent in some to the specification standards. Equipment manufacturers should specify the grade of agent required. Where an agent has been recycled/reclaimed for re-filling a fire protection system, its quality should be returned to no less than the minimum specifications prescribed by the equipment manufacturer. It is also prudent to understand what impurities are present to evaluate if it is suitable for use.

It is the responsibility of the testing laboratory to ensure that they keep abreast of any changes to standard specifications, including changes to any of the testing methods prescribed in the standards. Where laboratories are unable to follow the prescribed method in its entirety, any amended or alternative methods used need to be validated and verified that they are able to produce an equivalent result to the original method specified by the standard. Evidence of the validation and verification performed must be retained by the laboratory.

Persons in the supply chain who are responsible for recovering, reclaiming, re-filling and re-supplying halon and other halogenated gaseous fire extinguishing agents back into the supply chain should have access to a laboratory whether in house or externally, with the technical competence to perform to acceptable industry standards.

The technical competence of the laboratory depends on factors such as having suitable testing facilities; qualified/experienced personnel; access to the appropriate calibrated and maintained equipment; quality assurance processes/systems that ensure appropriate sampling and testing procedures/methods are used; the traceability of measurements to national standards; and accurate record keeping and reporting processes.

Either before, during, or after processing/treatment of the agents, batches can be subject to quality control measures to determine the condition of the agent prior to treatment as well as the effectiveness of the treatment process in returning the agent back to specification.

Laboratories with third party accreditations bear some type of symbol or endorsement indicating their accreditation to recognized standards. These laboratories are subject to independent evaluations of their processes and systems ensuring impartiality and competence. The laboratory may hold an accreditation; however, it may not necessarily have the accreditation for the specific agent tests. Where available, participants in the agent re-supply chain must use laboratories that are accredited to test the specific fire extinguishing agents.

Where there is use of laboratories without accreditation, the onus is always on the supplier of the agent to ensure that the laboratory has systems in place that are robust, and results supplied are accurate and reliable.
4.0 Agent Contamination

The presence of agents of questionable purity is an insidious problem that does not become apparent until an end user discharges an extinguisher or extinguishing system, often in a serious life safety or potential property loss setting. With an impure agent, the performance can range from poor or no fire extinguishing effectiveness to one where the impure agent may actually intensify the fire in the case where the impurity is a flammable material.

Generally speaking, end users do not have the means to confirm the purity of replenishment agents they have employed in fire extinguishers or in extinguishing systems. Instead, they have had to rely on the aftermarket supply chain to collect, process, test, and certify that the agent is of acceptable purity. From the end user’s perspective, it is that last step – the certification – that has been the ultimate basis for acceptance of the agent. Given that there has been at least one instance where certification documents were falsified by the agent supplier, it would seem that relying on a supplier’s certification alone can introduce risk with respect to agent purity. It is strongly recommended the end user demand a copy of the signed certification of the results for the halon used to fill the fire protection system or extinguishers and where available results are certified by an accredited laboratory.

To understand how and/or why agents with impurities can be supplied to end users, one has to look at the circumstances under which the impurities can be introduced. For all practical purposes, the impurities are usually introduced into the agent in four different manners. First, the impurities could already be present in the agent when the recycler or servicing company received the agent or the extinguisher containing the agent from an end user or intermediary. Second, the agent could become contaminated during processing by the recycler or servicing company when “good agent” is accidentally batched together with contaminated agent, thus causing the entire batch to become contaminated. This is referred to as ‘cross contamination with other halogenated chemicals.’ Third, the failure to adequately deep vacuum the equipment when changing from processing a different agent or refrigerant will cause the introduction of impurities by cross contamination with other halogenated chemicals or the introduction of other contaminants including oil, moisture, particulates or acids. Finally, agent that has been reclaimed to a standard can still become contaminated if it is put into cylinders or long-term storage tanks that have not been properly cleaned, and which contain contaminants such as water, oil and particulates.
5.0 The Agent Supply Chain

Figure 5.1 illustrates the parties involved in the supply chain for recycled halons and other gaseous halogenated fire extinguishing agents

![Figure 5.1 - Agent Supply Chain](image)

In the trading of recycled halons and other halogenated agents, there are usually five parties involved in commercial transactions:

- The **current end user** of the agent or extinguisher sells the contents of its extinguishers or extinguishing systems. This sale is usually to either a **servicing company** (a) or to a **recycling company** (b).

- The recycling company buys the agent or extinguisher for processing the agent and returning it to the required purity level and other testing requirements. The recycling company could buy the agent from the current end user (b) or from a servicing company (c).

- After processing the agent, the recycling company has the agent purity confirmed by an accredited **testing laboratory** (d to e). This laboratory is often a third party organisation and in other cases it is part of the recycling company.

- In most cases the recycling company sells the recycled agent to a servicing company (f) for use in recharging the extinguishers of a **new end user** (g). In some cases, the current end user and the new end user could be the same with the agent being processed and then banked (stored) for the end user. The banking service is sometimes provided by the recycling company and in other cases by the servicing company or by the new end user itself.
• Although the recycling company should provide complete certified test reports with each
tank or batch of agent sold, from time to time the new end user may deal with the testing
laboratory directly to have a sampling of its extinguishers contents tested to confirm that
the agent therein is up to specification (h to i). In some cases, the new end user employs
the servicing company as an intermediary with the testing laboratory.

There are instances where the services of a recycling company and a certified testing laboratory
are not part of the process. That is when the servicing company merely recovers agent from the
extinguishers or extinguishing systems of a current end user and then reuses that agent to
recharge the extinguishers of a new end user (steps a and g) with little or no purification efforts
and no testing. This is considered poor practice because one is never certain about either the
contents or the purity level in the current owner’s extinguisher and there is no provision to
identify any contamination introduced in the transfer process. Thus, extinguishers and
extinguishing systems recharged with agent by this simple method have no credibility with
regard to meeting purity or testing standards and thus performance effectiveness may be
compromized. In many nations, this practice is prohibited by law or regulation.

In the case of halon 1301, recycling companies often use a ‘halon identifier’ instrument [18] to
determine the percentage purity of newly received halon in order to prevent contamination of
other halon when combined in a batch. The halon identifier is an instrument that only provides an
indication of the primary gas concentration and does not provide the true level of purity that a
gas chromatograph (GC) or GC/Mass Spectrometer (MS) testing of the halon would provide
under laboratory conditions.

Depending on the purity of the agent when received for recycling, reclamtion efforts by the
recycling company may be as simple as nitrogen separation and filtration. If, however,
contamination with other halogenated agents or volatile residues is found, then the agent mixture
must be submitted to a distillation process to return the agent to a condition meeting the
requirements of the appropriate standard. In some cases, it may not be economically feasible to
“clean” the agent depending on the type and degree of contamination. "If the agent cannot be
feasibly returned to an acceptable certified purity level through distillation, then the agent should
be destroyed using a Montreal Protocol approved destruction technology. HTOC Technical Note
#5, Revision 2 – Destruction Technologies for Halons and Other Halogenated Gaseous Fire
Extinguishing Agents provides the latest information on destruction technologies suitable for
halons and other gaseous fire extinguishing agents [19]. Destroying contaminated halons and
other agents will ensure that these agents are removed from the supply chain and are not
available to inadvertently penetrate potentially useable stocks that do meet quality specifications.
6.0 Agent Contamination Mitigation Strategies

In reviewing the supply chain for recycled fire extinguishing agents, it is clear that the minimum mitigation strategies that can be employed to ensure that the agent meets an industry accepted quality standard are:

- **By the Recycler**: Employing robust quality assurance procedures that provide for (1) testing incoming agent to ensure that it is not contaminated before it is combined with other batched agents during the recycling process; (2) processing the batched agent in a manner to remove all contaminates to the specified levels, and (3) ensuring that no new contaminants can be introduced into the processed agent up through and including its final storage condition (cylinders, long-term storage tanks, drums, etc.) HTOC Technical Note # 2 on Emission Reduction Strategies [20] strongly reiterates the importance of having the correct, fit for purpose, maintained recovery/reclamation/transfer equipment. Competent and trained persons should be used to operate the equipment in such a way that the potential for contamination or loss of agent is avoided and mitigated as far as possible. HTOC Technical Note #2, Revision 3 also strongly recommends that any contaminated halon that cannot be recycled/reclaimed to acceptable purity levels be destroyed and not permitted to re-enter the supply chain.

- **By the Accredited Testing Laboratory**: In accordance with good laboratory practice, perform an analysis on samples of the recycled agent for each individual storage container (cylinder, drum, etc.) and provide written certification that the agent meets the required specification(s). Accredited laboratories are independently audited by a recognized third party certification body. See Table 6.1 for a list of laboratories that can be considered for performing testing and certification.

- **By the Servicing Company**: Preparing and following established good practices when recharging extinguishers and extinguishing systems to ensure that no contaminants are introduced at this stage either by the agent transfer equipment or by improper cleaning and drying of the extinguisher cylinder or systems.

- **By the New End User**: Periodically removing extinguishers from service and having the contents analyzed by a testing laboratory to check for contaminants in the contents. This can be done in a cost-effective manner by applying standard statistical sampling methods.
Table 6.1: Testing and Certification Laboratories

<table>
<thead>
<tr>
<th>Organization</th>
<th>Address</th>
<th>Phone</th>
<th>Fax</th>
<th>Website</th>
</tr>
</thead>
<tbody>
<tr>
<td>A-Gas Australia (3)</td>
<td>9-11 Oxford Road</td>
<td>+61 3 9368 9222</td>
<td>+613 9368 9233</td>
<td><a href="https://www.agasaustralia.com">https://www.agasaustralia.com</a></td>
</tr>
<tr>
<td></td>
<td>Laverton North, Victoria 3026</td>
<td></td>
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<tr>
<td></td>
<td>Australia</td>
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<td></td>
<td>Website: <a href="https://www.agasaustralia.com">https://www.agasaustralia.com</a></td>
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<tr>
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<td>One Blue Hill Plaza Pearl River</td>
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<td></td>
<td>New York, NY 10965, USA</td>
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<td></td>
<td>Phone: +1 845 735 6000</td>
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<td></td>
<td>Website: <a href="http://www.hudsontech.com">http://www.hudsontech.com</a></td>
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<tr>
<td>Intertek ETL Semko (2)</td>
<td>1717 Arlingate Lane</td>
<td>+1 614 279 8090</td>
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<td></td>
<td>Columbus, Ohio 43228</td>
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<td></td>
<td>Website: <a href="http://www.intertek.com/hvac/refrigerants/halon-analysis">http://www.intertek.com/hvac/refrigerants/halon-analysis</a></td>
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<tr>
<td>Kansai Gas Center</td>
<td>Murotani 2-1-3, Nishi-ku, Kobe, 651-2241 Hyogo</td>
<td>+81 78 991 7839</td>
<td>+81 78 991 7840</td>
<td><a href="http://www.n-eco.co.jp">http://www.n-eco.co.jp</a></td>
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<td>Japan</td>
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<td>Phone: +81 78 991 7840</td>
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<tr>
<td>National Refrigerants Laboratory (1)</td>
<td>Inc. 661 Kenyon Avenue</td>
<td>+1 800 262 0012</td>
<td>+1 856 455 2776</td>
<td><a href="http://www.refrigerants.com">http://www.refrigerants.com</a></td>
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<tr>
<td></td>
<td>Bridgeton, NJ 08302, USA</td>
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<td></td>
<td>Phone: +1 800 262 0012</td>
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<td>Phone: +1 856 455 2776</td>
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<td>Website: <a href="http://www.refrigerants.com">http://www.refrigerants.com</a></td>
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## Table 6.1: Testing and Certification Laboratories

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<th>Laboratory</th>
<th>Address</th>
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<th>Fax</th>
<th>Website</th>
</tr>
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<tbody>
<tr>
<td>NIPPON EKITAN Corporation</td>
<td>Kanto Gas Center, Kiyoku-cho 1-2, Kuki, 346-0035 Saitama Japan</td>
<td>+81 480 23 1313</td>
<td>+81 480 23 1329</td>
<td></td>
</tr>
<tr>
<td>Pan Gulf Environnemental Solutions (1)</td>
<td>P.O. Box 11175, Dammam 31453, Saudi Arabia</td>
<td>+966 13 812 86 72/72</td>
<td>+966 13 812 8671</td>
<td><a href="http://www.pgesolutions.com">www.pgesolutions.com</a></td>
</tr>
</tbody>
</table>

Note 1: These laboratories are AHRI (Air-Conditioning, Heating and Refrigeration Institute) certified to analyze refrigerant products, and because of product similarity, are also acceptable to the U.S. Department of Defense (DOD) for halon analysis.

Note 2: Although not identified as a certified laboratory, the U.S. DOD has utilized and accepted analyses provided by this laboratory.

Note 3: This laboratory is certified under ISO 17025 National Association of Testing Laboratories (NATA) in Australia.
7.0 References


3. GOST 15899-93, Specification for 1,1,2,2-tetrafluorodibromomethane (R-114B2), Gosstandart, Minsk, Republic of Belarus, https://gosstandart.gov.by


18. One such instrument is shown at http://www.refrigerantid.com/halon/identifier.html.


8.0  List of Acronyms and Abbreviations

AHRI  Air-Conditioning, Heating & Refrigeration Institute
ASTM  American Society for Testing and Materials International
DOD   U.S. Department of Defense
COP   Code of Practice
FK    Fluoroketone
GOST  Gosudarstvennye Standarty State Standard Gost
GWP   Global Warming Potential
HCFC  Hydrochlorofluorocarbon
HFC   Hydrofluorocarbon
HTOC  Halons Technical Options Committee
ISO   International Organization for Standardization
NATA  National Association of Testing Laboratories
NFPA  National Fire Protection Association
ODS   Ozone Depleting Substance
OOS   Out of Specification
PFC   Perfluorocarbon
TEAP  Technology and Economic Assessment Panel
UK    United Kingdom
UNEP  United Nations Environment Programme
U.S.  United States