

SEMI G84-0303

SPECIFICATION FOR STRIP MAP PROTOCOL

This specification was technically approved by the Global Automated Test Equipment Committee and is the direct responsibility of the North American Automated Test Equipment Committee. Current edition approved by the North American Regional Standards Committee on October 25, 2002 and November 22, 2002. Initially available at www.semi.org January 2003; to be published March 2003. Originally published November 2001.

1 Purpose

1.1 The purpose of this specification is to provide definition that will enable the implementation of strip mapping throughout the assembly and test process. This document will enable standardization of the strip map format and execution method throughout the factory floor.

1.2 This document describes the format and methods in which strip map information shall be formatted and used to transfer information about a strip to and from equipment using the SECS/GEM interface standard (see SEMI E5).

2 Scope

2.1 This specification will identify the messages within the SECS/GEM message set used in the implementation of strip mapping.

2.2 This specification will identify the Map Data Items (MDI) that are required or optionally may be used in the implementation of strip mapping.

2.3 This document will provide basic application notes to aid in the proper implementation of strip mapping.

2.4 This document will not address application or implementation for wafer test process.

NOTICE: This standard does not purport to address safety issues, if any, associated with its use. It is the responsibility of the users of this standard to establish appropriate safety and health practices and determine the applicability of regulatory or other limitations prior to use.

3 Limitations

3.1 This document does not address the transmission, file naming conventions, storage or archiving of strip maps.

4 Referenced Standards

4.1 SEMI Standards

SEMI E5 — SEMI Equipment Communications Standard 2 Message Content (SECS-II)

SEMI G81 — Specification for Map Data Items

SEMI T9 — Specification for Marking of Metal Lead-Frame Strips with a Two-Dimensional Data Matrix Symbol

NOTICE: Unless otherwise indicated, all documents cited shall be the latest published versions.

5 Terminology

5.1 Definitions

5.1.1 *assembly site* — the sub-contractor, supplier's facility or department that will be responsible for the assembly of IC devices.

5.1.2 *bottom-side* — the bottom of the strip as defined by the customer based on the customer master manufacturing drawing.

5.1.3 *cell* — a term used to represent the mapping item on a strip—unit, package, device, multi-chip module, etc.

5.1.4 *strip* — a leadframe, board, panel or other container which hold locations for semiconductor devices to be manufactured upon.

5.1.5 *strip map* — a record of data in a file that contains quality and historical information about each individual strip in a manufacturers lot.

5.1.6 *substrate* — a board or panel containing locations for semiconductor devices to be manufactured upon. Also referred to as strip, board or PCB.

5.1.7 *supplier* — a supplier of inspected/tested substrates (strips) and strip maps to the assembly site.

5.1.8 *top-side* — the top of the strip as defined by the customer based on the customer master manufacturing drawing.

6 Ordering Information

None.

7 Convention

7.1 The following conventions are used to represent data format. Each format is represented by an alphabetic character and optional data size given by "byte" unit. Exceptions are "*" and "List of". Refer to SEMI E5 for details.

7.1.1 A — ASCII character(s), may not be required terminal character such as null (00₁₆)

7.1.2 B — Binary

7.1.3 I — Integer number

7.1.4 U — Unsigned Integer number

7.1.5 List of — Combined item format consisting of one or more items; array (items of the same type) or structure (items of different types)

7.1.6 * — Any format as above or those allowed in SEMI E5.

8 Object Definitions

8.1 The implementation of Strip Mapping defines one standard object, the Strip Map.

8.1.1 *Strip Map Object Definition* — The Strip Map is a dynamic object created and modified through the assembly and test process. Each Strip Map Object is a logical representation of a manufacturing lead frame or substrate and maintains the current processing state for the group of die attached to that physical leadframe or substrate. Each Strip Map Object uniquely identifies a manufacturing leadframe or substrate using the ObjID (StripID) object attribute. The object attribute notation used in Table 1 is described in Conventions, Section 7.

9 Requirements

9.1 Equipment Communication

9.1.1 Communication of strip maps between host and equipment shall use the SECS I or HSMS protocols. Otherwise, some other general protocols, which are widespread in the data communication field, could be used as an alternative. However, these are not covered in this specification.

9.2 Map Data Items

9.2.1 The Standard Map Data Items in Table 1 contain the standard data items that shall be used in all strip mapping implementations.

9.2.2 The Strip Map Data Items in Table 2 are the data items and object attributes that will be used in the implementation of strip mapping via the SECS/GEM interface as needed by each equipment type and process step.

Table 1 Standard Data Item/Object Description and Settings

<i>Data Item</i>	<i>Description / Settings</i>	<i>Format</i>
SMORLOC	0 = Error 1 = Upper right 2 = Upper left 3 = Lower left 4 = Lower right > 4 = Error 5-63 Reserved	U1
ERRCODE	Code identifying an error	U2
ERRTEXT	Text string describing the error noted in the corresponding ERRCODE. Limited to 80 characters maximum.	A
OBJACK	Acknowledge Code	U1
ATTRID	Object attribute ID	A
ATTRDATA	Object attribute data	*
ACKC6	Strip map acknowledge 0 = successful 1 = failed	B

<i>Data Item</i>	<i>Description / Settings</i>	<i>Format</i>
OBJSPEC	A text string that has an internal format and that is used to point to a specific object instance. The string is formed out of a sequence of formatted substrings, each specifying an object's type and identifier. The substring format has the following four fields: object type, colon character ":", object identifier, greater than symbol ">", where the ":" is used to terminate an object type and the ">" is used to terminate an identifier field. The object type may be omitted where it may be otherwise determined. The final ">" is optional. The OBJSPEC for the StripMap Object is a zero-length item. No specifier required.	A
OBJTYPE	Identifier for a group or class of objects. All objects of this type must have the same set of attributes. Available object types: "StripMap"	A
OBJID	Object Identifier	A

Table 2 Strip Map Data Item/Object Description and Settings

<i>Attribute Name</i>	<i>Definition</i>	<i>Access</i>	<i>Required</i>	<i>Format</i>
ObjType	The object type, "StripMap"	RO	Y	A = "StripMap"
ObjID	The object. This is the StripID, FrameID or equivalent.	RO	Y	A
CellStatus	Cell Status is an array of values indicating the value of each unit/package/device on a strip. 0 = Functional Good Cell; Good die placed on Good cell. 1 = Unknown Cell Status. 2 = Defect/Non-functional Cell; the cell is marked as defect by previous steps. CellStatus shall be sent in an array of size Rows × Columns in row major format with respect to the OriginLocation. CellStatus may be provided by the host to the equipment as well as from the equipment to the host. Used in: Strip Map Upload and Strip Map Download	RW	Y' Either CellStatus or CellGrade or both are required if mapping is to be used.	U1
CellGrade	CellGrade is an array of values indicating the classification of each unit/package/device/cell on a strip. The process and equipment using a 'grading' scheme determine this classification and the associated values. Examples of a CellGrade are test bins, sort grades, package grade, marking grades etc. CellGrade shall be sent in an array of size Rows × Columns in row major format with respect to the OriginLocation.	RW	Y' Either CellStatus or CellGrade or both are required if mapping is to be used.	U1
Columns	The number of columns of (measured) device matrix on substrate, that is the number of devices on the X-axis of the map data. It is required if the type of the map data is simple array structure.	RO	Y	U4

<i>Attribute Name</i>	<i>Definition</i>	<i>Access</i>	<i>Required</i>	<i>Format</i>
OriginLocation	<p>Coordinate system on substrate to address devices. Location of the datum point or the origin of the coordinates shall be one of the following:</p> <p>0 = Not currently supported 1 = Upper right – top side 2 = Upper left – top side 3 = Lower left – top side* 4 = Lower right – top side 5 = Not currently supported 6 = Upper right – bottom side 7 = Upper left – bottom side 8 = Lower left – bottom side 9 = Lower right – bottom side</p> <p>* Default value is 3 (=Lower left – top side) if this item is not present.</p> <p>Typically Used in: Strip Map Upload and Strip Map Download</p>	RO	Y	U1
Rows	The number of rows of (measured) device matrix on substrate, that is the number of devices on the Y-axis of the map data. It is required if the type of the map data is simple array structure.	RO	Y	U4
SubstrateID	A character code that may be printed or encoded on the substrate, which uniquely identifies the substrate, and can be used to correlate the map with physical substrate. SubstrateID is either StripID or FrameID.	RW	Y	A
BinGrade	<p>A BinCode (see SEMI G81) is a value describing the grading of a die. BinGrade is an array of bin codes used to describe each die on the wafer.</p> <p>BinCodeList shall be sent in an array size Rows x Columns in row major format with respect to the OriginLocation.</p> <p>Typically used in: Strip Map Upload</p>	RW	N	U1
DefectCode	<p>DefectCodes are a set of codes representing defects that are detected at each process step by equipment or a human.</p> <p>DefectCodes shall be sent in an array of size Rows x Columns in row major format with respect to the OriginLocation.</p> <p>If there is no defect, the DefectCode is 0 (Zero)</p> <p>Typically Used in: Strip Map Upload and Strip Map Download</p>	RW	N	U1 or A
LotID	Production lot identifier for this data.	RW	N	A

<i>Attribute Name</i>	<i>Definition</i>	<i>Access</i>	<i>Required</i>	<i>Format</i>
ContainerID	A character code that may be printed or encoded on the substrate carrier, which uniquely identifies the substrate carrier. ContainerID can be MagazineID, CarrierID or CassetteID.	RW	N	A
WaferID	A character code that may be printed or encoded on the wafer, which uniquely identifies the wafer. Used when equipment reports wafer id with or in parallel with strip map data.	RW	N	A
XYPosition	<p>XYPosition represents the X and Y coordinate position of the die on the wafer. XYPosition must be in (X,Y) order multi-item and shall be made up of data items DieX and DieY (See SEMI G81).</p> <p>For each XYPosition a WaferID is given.</p> <p>This information is used when wafer id and die location on the wafer is to be associated with the strip id and location of the unit/package/device/cell on a strip.</p> <p>XYPosition is set to 32767, 32767 when a strip is removed from the equipment. This represents no wafer die information.</p> <p>Valid range for X and Y is -32768 .. 32767</p>	RW	N	I2

9.3 Map Data Protocol

9.3.1 The strip mapping standard implementation shall use the SECS/GEM standard Object Services (S14,F1/F2) and standard Collection Event S6,F11/F12 Streams and Functions for the transfer of Strip Maps to and from equipment (see SEMI E5).

9.3.2 The use of object services will allow the equipment to request the type of information from the host with predefined attributes. The host will then reply with the list of attributes requested. This will enable application specific behavior of the mapping process by equipment types.

9.3.3 The use of the Collection Event Streams and Functions will allow event-based communication from the equipment to the host on the status of the strip mapping process.

9.3.4 Strip Map Download

9.3.4.1 The equipment shall request a strip map

download from the host using the Standard Object Services (OSS) command, S14,F1 GetAttribute Request (GAR). Please refer to Section 9.2.1.

9.3.4.2 The equipment shall include all mandatory data attributes in GAR request as well as any optional information required by the particular equipment to implement the strip mapping capability.

9.3.4.3 The host will respond to the GetAttribute Request with an S14,F2, Get Attribute Data (GAD).

9.3.5 Strip Map Upload

9.3.5.1 The equipment will provide updates to the strip map via the S6,F11 event.

9.3.5.2 All mandatory data items are required to be included in the upload event along with any optional equipment specific data items. Please refer to Section 9.2.2 for examples.

10 Description

10.1 See SEMI G81 for further definitions of the strip map required items.

10.2 *Strip Map Required Attributes*

10.2.1 The following set of Data Items are mandatory in the uploads and downloads scenarios for strip maps.

- Strip ID
- Rows
- Columns
- OriginLocation
- CellStatus | CellGrade

10.2.2 Each equipment shall have an attribute for the product or strip type that will describe the standards process direction and physical origin of the strip. If the physical origin and the factory defined origin are not the same, it is the responsibility of the equipment to perform the appropriate translations to maintain integrity of the strip map information.

10.3 *Strip Map Data Protocol Examples*

10.3.1 *Strip Map Download (Example)*

S14,F1 GetAttr Request (GAR) S,H<-E

10.3.1.1 Description: This message is used to request a set of specified attributes for one or more objects.

Structure: L,5

1. <OBJSPEC = '>
2. <OBJTYPE = 'StripMap'>
3. L,1
 1. <OBJID1 = StripID = 'A1000001'>
4. L,0 (always zero list, no filters defined)
5. L,5 (any length and order of data items)
 1. <ATTRID = OriginLocation >
 2. <ATTRID = 'Rows'>
 3. <ATTRID = 'Columns'>
 4. <ATTRID = 'CellStatus'>
 5. <ATTRID = 'LotID'>

NOTE: This is an example only. Each equipment must request all mandatory data items and can additionally request any optional data items that it needs to fully implement strip mapping capability.

S14,F2 GetAttr Data (GAD) M,H->E

10.3.1.2 Description: This message is used to transfer the set of requested attributes for the specified object(s). The order of attributes is retained from the primary message.

Structure: L,2

1. L, 1
 1. L,2
 1. <OBJID = StripID = 'A1000001'>
 2. L,5
 1. L,2
 1. <ATTRID = 'OriginLocation' >
 2. <ATTRDATA = '0'>
 2. L,2
 1. <ATTRID = ' Rows'>
 2. <ATTRDATA = 6>



3. L,2
 1. <ATTRID = ' Columns '>
 2. <ATTRDATA = 24>
4. L,2
 1. <ATTRID = ' CellStatus'>
 2. <ATTRDATA = 6>
5. L,2
 1. <ATTRID = ' LotID '>
 2. <ATTRDATA = 'Lot1>
2. L,2
 1. <OBJACK = 0>
 2. L,0

10.3.2 Strip Map Upload (Example)

S6,F11 Event Report M, H<-E

10.3.2.1 Description: This message is used to report the current map status. This event will occur at the end of a strip as well as at any point during the processing of the strip when the equipment enters a condition where an operator may have the opportunity to handle the strip or the equipment has an opportunity for error. This event shall always be sent with the current status of the map at the time the equipment gets changed from local to remote.

10.3.2.1.1 For a description of filters, see S6,F11.

Structure: L,3

1. <DATAID>
2. <CEID>
3. L,1
 1. L,2
 1. <RPTID>
 2. <L,n
 1. <xxxx>
 - n. <yyyy>

S6,F12 Event Report Acknowledge H->E

Description: Acknowledge or Error

Structure: <ACKC6>

11 Application Scenarios

11.1 Strip Map Download

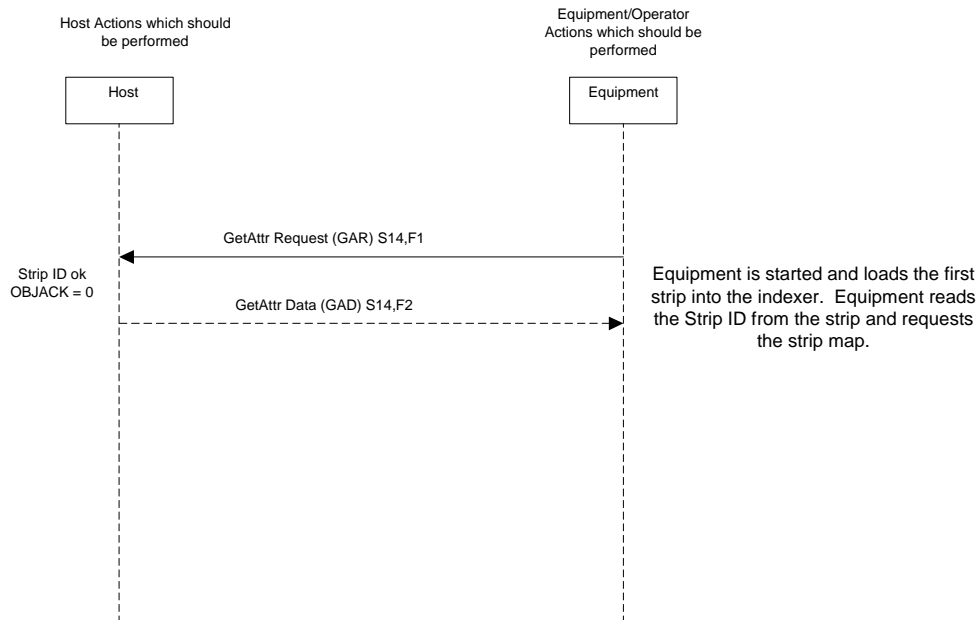


Figure 1
Strip Map Download

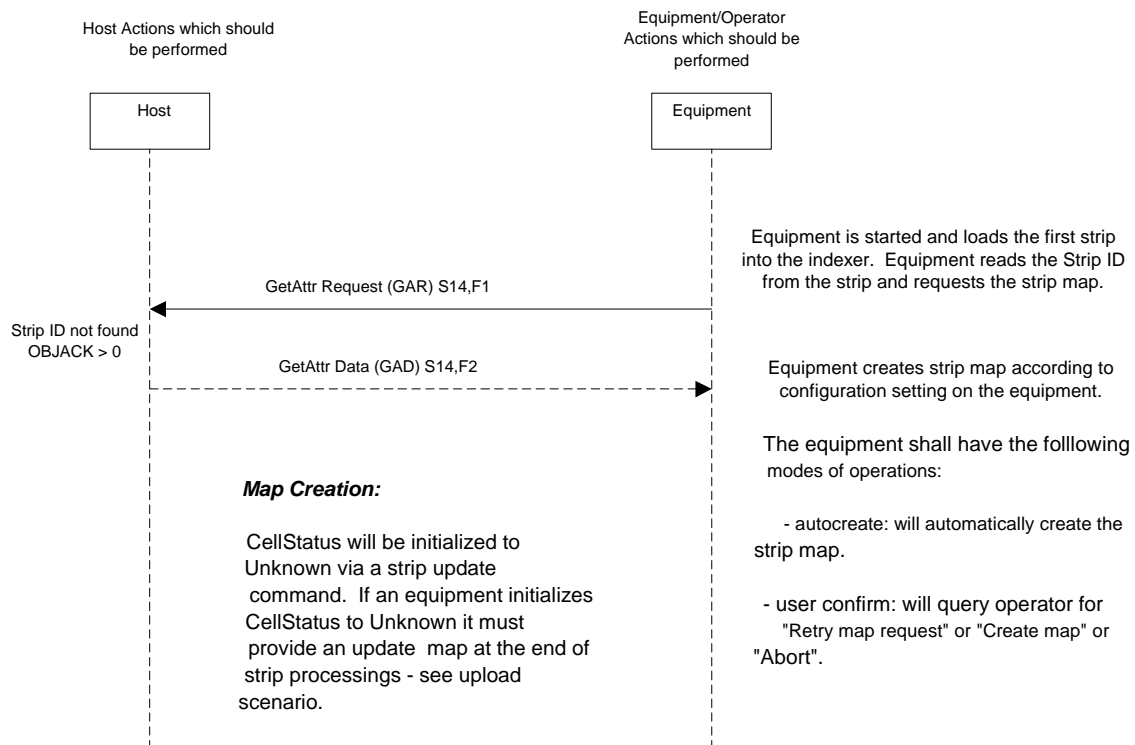


Figure 2
Strip Map Create

11.1.1 A typical equipment scenario is that for each strip arriving at the equipment input, a strip map request (S14,F1) and subsequent download (S14,F2) will occur. See Figure 1. Depending on the equipment type, there may be some variations on the optional attributes requested, the timing of events and exception handling.

11.2 Strip Map Creation

11.2.1 A map must exist before it can be sent to an equipment type. Some equipment may be required to support the creating of the map for the first time. See Figure 2. For these equipment two modes are identified, “autocreate” and “user confirm”. To use these modes the equipment shall behave as indicated below.

11.2.2 If equipment supports an “autocreate” mode the equipment will process the strip as normal when a new strip arrives. The equipment will assume each location on the strip has an unknown status. Should the

equipment be required to send a strip map during processing due to equipment errors, the strip map shall contain the unknown CellStatus value whenever the status is unknown. All other CellStatus indicators shall be “Good” or “Bad”. See Upload scenario.

11.2.3 If equipment supports a “user confirm” mode, the equipment shall request operator intervention to decide how to deal with a missing strip map. The options are “Create map” or “Abort”. If the operator selects “Create map” the above scenario will be valid. If the operator selects “Abort”, the equipment shall send an alarm to the host and not process the strip. The strip will not be processed unless the equipment is removed from the “strip map” mode.

11.3 Strip Map Upload

11.3.1 For equipment with the capability of detecting defective units on a strip, a strip map upload is required. The upload will report new definitions of each “cell”.

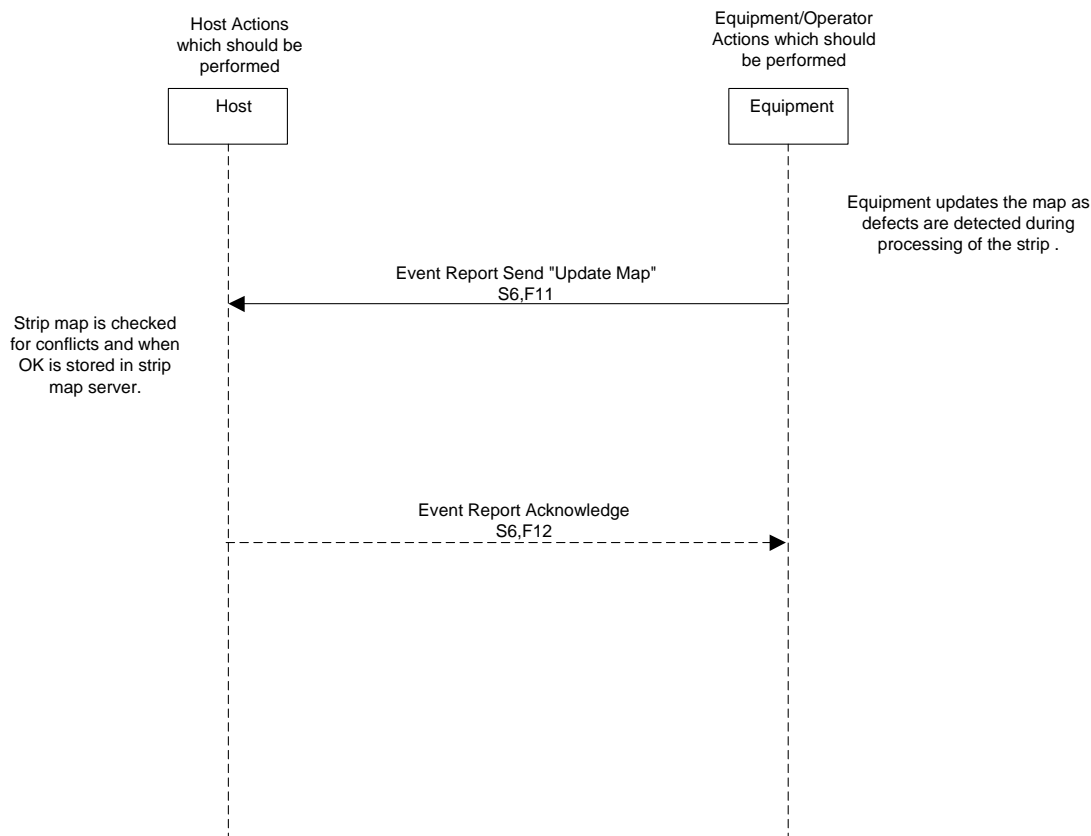


Figure 3
“Update Map” Event

11.3.1.1 An Event Report “Update Map” will be sent if new defects have been detected and not previously logged to the host for the following conditions. See Figure 3.

1. At the end of processing current strip if new defects have been detected
2. Whenever the equipment changes state from Remote to Local
3. Whenever the equipment interlocks are triggered
4. Whenever the equipment stops and requires operator intervention.

11.3.2 Unsuccessful Transaction

11.3.2.1 Equipment will stop and signal an alarm to the host if the equipment detects that a strip map has not been successfully uploaded to the host.

11.3.2.2 When possible, the equipment shall support intervention by the Operator with the following corrective actions:

1. “retry upload”
2. “abort upload”

11.3.2.3 If the “retry upload” is selected the Upload Event will be resent to the host.

11.3.2.4 If “abort” is selected by the operator an appropriate Event shall be sent with the strip map ID to indicate to the host that the strip map has been aborted. The equipment will not process the strip unless the equipment is removed from “strip map” mode.

11.4 Strip End Event

11.4.1 At the end of each strip, a strip end event shall be sent to the host which indicates that the strip has successfully completed the activity at that equipment.

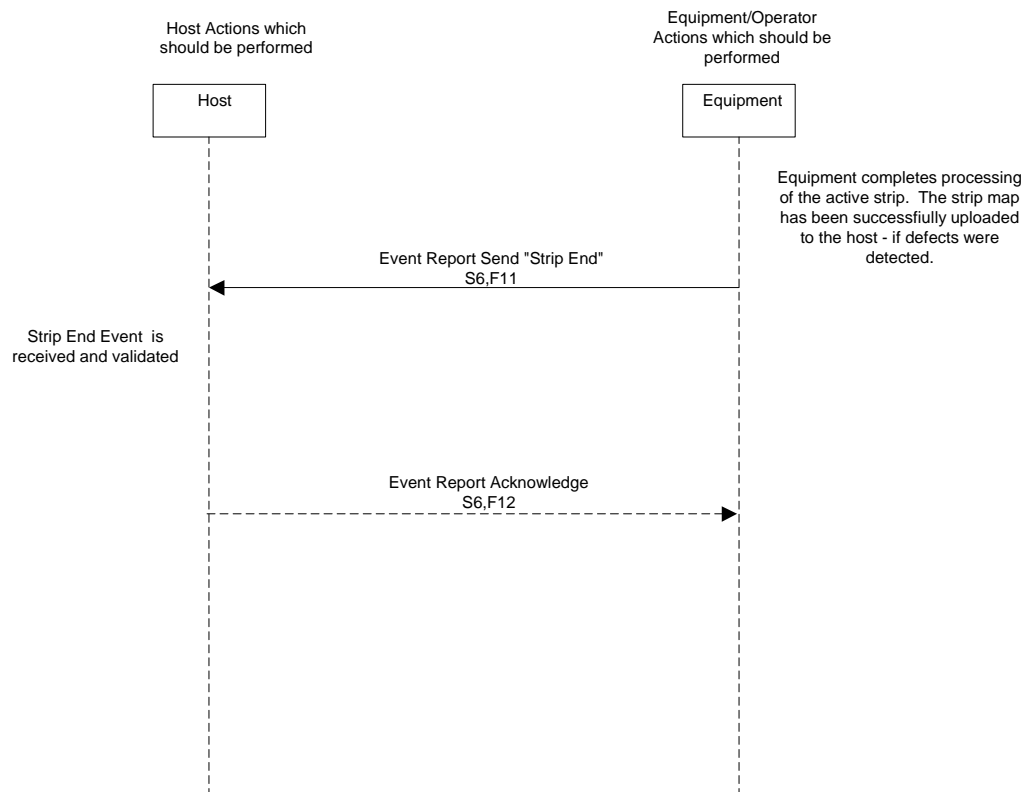


Figure 4
“Strip End” Event

11.5 *In Process Strip Map Related Errors*

11.5.1 Each equipment type shall define the type of errors that can occur during processing which could cause strip map integrity problems and provide documented scenarios of how the equipment will handle each situation.

11.5.2 Some potential erroneous situations:

- Equipment lost strip map while processing
- Strip is removed prior to completing processing strip
- Strip jams before being moved out of equipment—no Strip Map End Event occurred

APPENDIX 1 STRIP MAP

NOTICE: The material in this appendix is an official part of SEMI G84 and was approved by full letter ballot procedures on August 27, 2001.

A1-1 This appendix shows how the map data items may be applied to strips as well as some additional information specific to this substrate type.

A1-2 The StripID shall follow the SEMI T9 specification, when applicable, or may be user definable by the factory.

A1-3 For any given process there must be a way to ensure correct orientation of the strip. The end user is responsible for providing a master manufacturing drawing showing the top side of the strip. This drawing must show any special markings or patterns that are needed to reliably flip and rotate a physical strip until it is oriented the same as the drawing. Figure A1-1 defines what is meant by top side and hence bottom side. It also defines what is meant by upper, lower and left and right.

A1-4 Each strip type will have a factory defined value for OriginLocation. This is the origin reference for the row 1, column 1 location on the strip. The factory origin, OriginLocation is selected as one of four corners by the factory host system. The row and column index will be referenced from the selected reference. This information will be provided to the equipment by the host in each strip map download scenario.

- 1 = Upper right (UR) top side
- 2 = Upper left (UL) top side
- 3 = Lower left (LL) top side
- 4 = Lower right (LR) top side
- 6 = Upper right (UR) bottom side
- 7 = Upper left (UL) bottom side
- 8 = Lower left (LL) bottom side
- 9 = Lower right (LR) bottom side

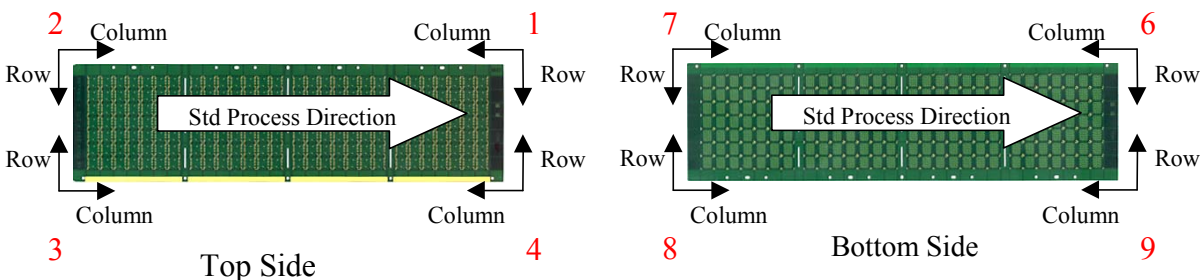


Figure A1-1
Factory Origin Settings

NOTICE: SEMI makes no warranties or representations as to the suitability of the standard set forth herein for any particular application. The determination of the suitability of the standard is solely the responsibility of the user. Users are cautioned to refer to manufacturer's instructions, product labels, product data sheets, and other relevant literature respecting any materials mentioned herein. These standards are subject to change without notice.

The user's attention is called to the possibility that compliance with this standard may require use of copyrighted material or of an invention covered by patent rights. By publication of this standard, SEMI takes no position respecting the validity of any patent rights or copyrights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of any such patent rights or copyrights, and the risk of infringement of such rights, are entirely their own responsibility.

SEMI G85-0703

SPECIFICATION FOR MAP DATA FORMAT

This specification was technically approved by the Global Automated Test Equipment Committee and is the direct responsibility of the North American Automated Test Equipment Committee. Current edition approved by the North American Regional Standards Committee on March 12, 2003 and April 11, 2003. Initially available at www.semi.org May 2003; to be published July 2003. Originally published November 2001; previously published March 2003.

1 Purpose

1.1 This document describes in detail how the data items that relate to electronic mapping are to be represented in a file format.

1.2 Although the examples given in this document are for wafers, the map data format described can be applied to any substrate including, but not limited to, wafer, tray, strip or tape.

2 Scope

2.1 This document applies to format of map data items. This document does not address the transmission, file naming conventions, storage or archiving of maps.

2.2 The semantics of the map data items are not specified in this document.

NOTICE: This standard does not purport to address safety issues, if any, associated with its use. It is the responsibility of the users of this standard to establish appropriate safety and health practices and determine the applicability of regulatory or other limitations prior to use.

3 Limitations

3.1 This document is limited to representing maps for MapType = "Array". See SEMI G81 for a definition of MapType.

4 Referenced Standards

4.1 SEMI Standards

SEMI G81 — Specification for Map Data Items

4.2 World Wide Web Consortium Documents¹

Extensible Markup Language (XML) 1.0 (Second Edition. Namespaces in XML)

NOTICE: Unless otherwise indicated, all documents cited shall be the latest published versions.

¹ World Wide Web Consortium Documents are available from Massachusetts Institute of Technology Laboratory for Computer Science, 200 Technology Square, Cambridge, MA 02139, USA Telephone: + 1.617.253.2613, Fax: + 1.617.258.5999, <http://www.w3.org/TR/REC-xml>

5 Terminology

5.1 Definition

5.1.1 *map* — a two dimensional array of bin codes derived from electrical test data of a two dimensional substrate including, but not limited to, wafer, tray, strip or tape.

6 Ordering Information

None.

7 Description

7.1 XML Notation Extensible Markup Language is the universal language for data on the Web. It gives developers the power to deliver structured data from a wide variety of applications to the desktop for local computation and presentation. XML allows the creation of unique data formats for specific applications. It is also an ideal format for server-to-server transfer of structured data.

7.2 XML consists of elements (referred to as 'items' in the remainder of this document) that may have attributes and nested items.

7.2.1 Example:

```
<Map xmlns: " http://www.semi.org" >
  <Device>
  </Device>
</Map>
```

defines a Map item with the attribute xmlns: "http://www.semi.org" which defines a namespace and the governing body for that namespace. This is in accordance with the "Namespaces in XML" specification (see references). Specifying the namespace ensures that the names within the Map element are distinguishable from the same names in other non-SEMI XML documents.

7.2.2 The Map item contains nested items called Device.

7.3 The listing below is an example of a well-formed map data file for a single map with all the map data items. A map supplier may insert their own elements

and nodes as long as the resulting document is well formed XML and the elements, nodes and attributes defined in SEMI G85 remain unaffected. The elements of this template will be described in the sections that follow. Examples of map data files with data are shown in the appendices.

```
<? xml version=" 1.0" ? >
<Map
  xmlns: " http://www.semi.org"
  SubstrateType=" "
  SubstrateId=" "
  FormatRevision=" " >
  <Device
    ProductId=" "
    LotId=" "
    Orientation=" "
    DeviceSizeX=" "
    DeviceSizeY=" "
    Rows=" "
    Columns=" "
    BinType=" "
    FrameId=" "
    NullBin=" "
    SupplierName=" "
    OriginLocation=" "
    CreateDate=" "
    LastModified=" "
    Status=" " >
    <ReferenceDevice
      ReferenceDeviceX=" "
      ReferenceDeviceY=" "
      RefDevicePosX=" "
      RefDevicePosY=" "
    />
    <Bin
      BinCode=" "
      BinQuality=" "
      BinDescription=" "
    />
    <Data MapName=" " MapVersion=" " >
      <Row><![CDATA[]]></Row>
    </Data>
  </Device>
</Map>
```

7.3.1 The order of attributes and sub items within their parent item is not significant but the parent child relationship is. One exception is the Row items which must match the actual rows on the substrate being mapped.

7.4 Every map file starts with the declaration:

```
<? xml version=" 1.0" ? >
```

This line informs the parser that the data that follows conforms to the XML Version 1.0 specification.

7.5 The data for each wafer is enclosed within the map data item:

```
<Map xmlns: http://www.semi.org
  SubstrateType=" Wafer"
  SubstrateId =" ZSDGS88DF"
</Map>
```

7.5.1 The attribute setting, xmlns: "http://www.semi.org" informs the parser that the item and attribute names within this item are unique within the namespace identified as SEMI.

7.5.2 All the attributes within the Map item are defined in the SEMI G81.

7.5.3 All the items within the Map Item are defined in this document.

7.5.4 Some of these attributes and items are mandatory and some are optional as defined in Table 1.

7.5.5 One or more maps can be represented in a single file by simply repeating the <Map> element. Each map can be distinguished based on an identifier attribute as shown in the following example:

```
<? xml version=" 1.0" ? >
<Maps>
  <Map xmlns: " http://www.semi.org"
    SubstrateType=" Wafer"
    SubstrateId =" ZSDGS88DF"
  </Map>
  <Map xmlns: " http://www.semi.org"
    SubstrateType=" Wafer"
    SubstrateId=" JDUJ102MJS"
  </Map>
</Maps>
```

7.6 The Map item contains one or more Device items. This allows multiple overlaying maps of different device types to be represented. The Device item contains a number of attributes and some sub items.

7.6.1 The ReferenceDevice item contains attributes which describe a single reference device. Several ReferenceDevice items may be represented by repeating this item with different attribute values.

7.6.2 The Bin item contains attributes which describe a single bin code. Several Bin items may be represented by repeating this item with different attribute values.

7.6.3 The Data item contains attributes and a list of Row items. Several Data items may be represented by repeating this item with different attribute values. This

might be used for example to represent versions of the map after each process step.

7.6.4 The Row items are defined as XML CDATA section. The example below shows the Data item for a binary map with two rows and two columns.

```
<Data>
  <Row><![CDATA[FFFF]]></Row>
  <Row><![CDATA[FF14]]></Row>
</Data>
```

7.6.4.1 A CDATA section contains data that should not be parsed by the XML parser. After <![CDATA[, only the string]]> will be interpreted by the XML parser. If this string occurs in the actual data (for example in an

ASCII map) then it must be replaced by the string]]> so that the XML parser will not interpret it as the end of the CDATA section.

7.6.4.2 Each Row item specifies the BinCode for each column in the row in a format that depends on the BinType attribute.

7.6.4.3 The Row items must match the actual rows on the substrate being mapped. The first row is the topmost row as viewed with the substrate oriented according to the value of Orientation.

7.6.4.4 The number of Row items must match the Rows attribute if specified.

7.6.4.5 The number of columns in each Row item must match the Columns attribute if specified.

Table 1 Items and Attributes Used

<i>Name</i>	<i>Item or Attribute</i>	<i>Defined In</i>	<i>Mandatory or Optional</i>	<i>Default Value</i>
xmlns	Attribute	Map Data Format	Mandatory	Must be "http://www.semi.org"
NullBin	Attribute	Map Data Items	Mandatory	No default
Map	Item	Map Data Format	Mandatory	No default
Device	Item	Map Data Format	Mandatory	No default
Data	Item	Map Data Format	Mandatory	No default
Row	Item	Map Data Format	Mandatory	No default
FormatRevision	Attribute	Map Data Items	Optional	Latest version of this standard
ProductId	Attribute	Map Data Items	Optional	""
LotId	Attribute	Map Data Items	Optional	""
Orientation	Attribute	Map Data Items	Mandatory	0
WaferSize	Attribute	Map Data Items	Optional	0
DeviceSizeX	Attribute	Map Data Items	Optional	0
DeviceSizeY	Attribute	Map Data Items	Optional	0
StepSizeX	Attribute	Map Data Items	Optional	0
StepSizeY	Attribute	Map Data Items	Optional	0
CassetteId MagazineId	Attribute	Map Data Items	Optional	""
Rows	Attribute	Map Data Items	Optional	Can be derived from number of Row items
Columns	Attribute	Map Data Items	Optional	Can be derived from Row items
BinType (See Note 3.)	Attribute	Map Data Items	Mandatory	"ASCII"
ReferenceDevice	Item	Map Data Format	Mandatory (See Note 1.)	No default
ReferenceDeviceX	Attribute	Map Data Items	Mandatory (See Note 1.)	No default
ReferenceDeviceY	Attribute	Map Data Items	Mandatory (see Note 1)	No default
RefDevicePosX	Attribute	Map Data Items	Optional	0.0
RefDevicePosY	Attribute	Map Data Items	Optional	0.0
MapName	Attribute	Map Data Items	Optional	""
MapVersion	Attribute	Map Data Items	Optional	""
BinCode	Attribute	Map Data Items	Optional	0
BinCount	Attribute	Map Data Items	Optional	0

<i>Name</i>	<i>Item or Attribute</i>	<i>Defined In</i>	<i>Mandatory or Optional</i>	<i>Default Value</i>
BinQuality	Attribute	Map Data Items	Optional	""
BinDescription	Attribute	Map Data Items	Optional	""
FrameId	Attribute	Map Data Items	Optional	""
SupplierName	Attribute	Map Data Items	Optional	""
OriginLocation	Attribute	Map Data Items	Mandatory	0 (Center topside)
CreateDate	Attribute	Map Data Items	Optional	""
Status	Attribute	Map Data Items	Optional	""
Bin	Item	Map Data Format	Optional	No default
SlotNumber	Attribute	Map Data Items	Optional	0
SubstrateNumber	Attribute	Map Data Items	Optional	0
GoodDevices	Attribute	Map Data Items	Optional	0
SubstrateType	Attribute	Map Data Items	Mandatory	
SubstrateId	Attribute	Map Data Items	Mandatory	
LastModified	Attribute	Map Data Items	Optional	

NOTE 1: There must be at least one ReferenceDevice item containing the ReferenceDeviceX and ReferenceDeviceY attributes.

NOTE 2: A substrate must have one and only one of these identifiers depending on the substrate type.

NOTE 3: Since XML is an ASCII only format BinType may only take the ASCII representation values "ASCII", "Decimal", "HexaDecimal", and **not** "Binary".

APPENDIX 1

EXAMPLE MAP DATA FILES²

NOTICE: The material in this appendix is an official part of SEMI G85 and was approved by full letter ballot procedures on March 12, 2003.

A1-1 Simple ASCII Wafer Map

A1.1 This example illustrates the simplest possible wafer map. It uses ASCII bin codes and contains only the required data items.

```
<? xml version=" 1.0" ? >
<Map xmlns: " http://www.semi.org"
SubstrateType=" Wafer"
SubstrateId=" ZSDGS88DF" >
<Device
  Orientation=" 0"
  BinType=" Ascii"
  NullBin=" "
  OriginLocation=" 1" >
<Data >
  <Row><! [CDATA[ 2C ]]></Row>
  <Row><! [CDATA[ ~ 2C2 ]]></Row>
  <Row><! [CDATA[222A1~ ]]></Row>
  <Row><! [CDATA[21~ C22]]></Row>
  <Row><! [CDATA[ ~ 2C2 ]]></Row>
  <Row><! [CDATA[ 2C ]]></Row>
</Data>
</Device>
</Map>
```

A1-2 Detailed Binary Wafer Map

A1-2.1 The example below illustrates the use of binary bin codes and contains not only the required data items, but also some data items provided by the supplier.

```
<? xml version=" 1.0" ? >
<Map xmlns: " http://www.semi.org"
SubstrateType=" Wafer"
SubstrateId=" ZSDGS88DF"
FormatRevision=" SEMI G85-0703" >
<Device
  ProductId=" 854CS1C"
  LotId=" wksfd87dcj37"
  Orientation=" 0"
  DeviceSizeX=" 343.8"
  DeviceSizeY=" 373.1"
  Rows=" 6"
  Columns=" 6"
  BinType=" HexaDecimal"
  FrameId=" KJKSDFK45"
  NullBin=" FF"
  SupplierName=" Company X"
  OriginLocation=" 1"
  CreateDate=" 2002110112000000"
  LastModified=" 2003010612000000"
  Status=" Product" >
<ReferenceDevice
  ReferenceDeviceX=" 2"
  ReferenceDeviceY=" -3"
  RefDevicePosX=" 2"
  RefDevicePosY=" 2"
/>
<ReferenceDevice
  ReferenceDeviceX=" 4"
  ReferenceDeviceY=" -5"
  RefDevicePosX=" 2"
  RefDevicePosY=" 2"
/>
<Bin
  BinCode=" 02"
  BinQuality=" Pass"
  BinDescription=" 500MHz"
/>
<Data MapName=" SortGrade" MapVersion=" 1" >
```

² These examples are for a wafer map identified by WaferId. Other substrate types may also be represented. See Table 1.

```
<Row><! [CDATA[FFFF020CFFFF]]></Row>
<Row><! [CDATA[FF14020C02FF]]></Row>
<Row><! [CDATA[0202020C0116]]></Row>
<Row><! [CDATA[0201140C0202]]></Row>
<Row><! [CDATA[FF14020C02FF]]></Row>
<Row><! [CDATA[FFFF020CFFFF]]></Row>
</Data>
</Device>
</Map>
```

A1-3 Simple Wafer Map with 2 Byte Bin Codes

A1-3.1 This example illustrates the simplest possible wafer map. It uses 2 byte bin codes and contains only the required data items.

```
<? xml version=" 1.0" ? >
<Map xmlns=" http://www.semi.org"
  SubstrateType=" Wafer"
  SubstrateId =" ZSDGS88DF" >
  <Device
    Orientation=" 0"
    BinType=" Integer2"
    NullBin=" 00FF"
    OriginLocation=" 1" >
    <Data >
      <Row><! [CDATA[00FF00FF0002000C00FF00FF]]></Row>
      <Row><! [CDATA[00FF00140002000C000200FF]]></Row>
      <Row><! [CDATA[000200020002000C00010016]]></Row>
      <Row><! [CDATA[000200010014000C00020002]]></Row>
      <Row><! [CDATA[00FF00140002000C000200FF]]></Row>
      <Row><! [CDATA[00FF00FF0002000C00FF00FF]]></Row>
    </Data>
  </Device>
</Map>
```

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SEMI G86-0303

TEST METHOD FOR MEASUREMENT OF CHIP (DIE) STRENGTH BY MEAN OF 3-POINT BENDING

This test method was technically approved by the Global Assembly and Packaging Committee and is the direct responsibility of the Japanese Packaging Committee. Current edition approved by the Japanese Regional Standards Committee on January 10, 2003. Initially available at www.semi.org January 2003; to be published March 2003.

1 Purpose

1.1 This test method defines a procedure for the evaluation of die strength by the mean of 3-point bending method.

2 Scope

2.1 This test method applies only for 3-point bending method, and other methods will be defined by the separate documents.

2.2 This test method is used to measure die strength for dies from processed wafers.

2.3 Wafer thinning technology becomes popular to meet the demand for thin packages, so the die strength data is critical for the die quality and certification. This standard is one of the documents, which describe the Die Strength Evaluation Method, Measurement Data Summary Technique and Data Usage for Test Report.

NOTICE: This standard does not purport to address safety issues, if any, associated with its use. It is the responsibility of the users of this standard to establish appropriate safety and health practices and determine the applicability of regulatory or other limitations prior to use.

3 Referenced Standards

3.1 ISO Standards¹

ISO 468: Surface roughness — Parameters, their values and general rules for specifying requirements

ISO/DIS 6508-1: Metallic materials — Rockwell hardness test (scales A,B,C,D,E,F,G,H,K,N,T) — Part 1: Test method

NOTICE: Unless otherwise indicated, all documents cited shall be the latest published versions.

4 Terminology

4.1 Definitions

4.1.1 *deflection* δ — distance over which the top or bottom surface of the test specimen at midspan deviates from its original position during flexure.

NOTE 1: It is expressed in millimeters (mm).

4.1.2 *flexural stress* σ_f — nominal stress of the outer surface of the test specimen at midspan.

NOTE 2: It is calculated from the relationship given in Section 8.1, equation (1) in Section 9.1, and is expressed in megapascals (Mpa).

4.1.3 *flexural stress at break* σ_{fB} — flexural stress at break of the test specimen.

NOTE 3: It is expressed in megapascals (Mpa).

4.1.4 *loading edge* — edge to apply the force to test specimen.

4.1.5 *speed of testing* v — rate of loading edge movement.

NOTE 4: It is expressed in millimeters per minute (mm/min).

4.1.6 *supports* — two supports to support the test specimen during flexural test.

5 Summary of Method

5.1 This test method is based on 3-point bending test and calculating the flexural stress at break.

6 Test Apparatus

6.1 Test Machine

6.1.1 The machine shall be capable of maintaining the test speed with the tolerance of ± 0.05 mm/min.

6.1.2 The machine shall be capable of measuring and recording the test force in newtons (N).

6.1.3 The error in the indicated force shall not exceed 1% of the actual value.

6.2 Supports and Loading Edge

6.2.1 Two supports and a central loading edge shall be arranged as shown in Figure 1.

¹ International Organization for Standardization, ISO Central Secretariat, 1, rue de Varembe, Case postale 56, CH-1211 Geneva 20, Switzerland. Telephone: 41.22.749.01.11; Fax: 41.22.733.34.30
Website: <http://www.iso.ch>

6.2.2 The radius of the supports and the loading edge shall be $0.3 \text{ mm} \pm 0.02 \text{ mm}$.

6.2.3 The width of supports B_1 and loading edge B_2 shall be longer than the width of test specimen b .

6.2.4 Two supports shall be parallel to X direction within $\pm 0.03 \text{ mm}$ and to Z direction within $\pm 0.02 \text{ mm}$. And two supports to the loading edge shall be parallel

to X direction within $\pm 0.03 \text{ mm}$ and to Z direction within $\pm 0.02 \text{ mm}$ (see Figure 2).

6.2.5 The tip of the supports and loading edge shall be hardened more than HRC60 specified in ISO/DIS 6508-1.

6.2.6 The tip of the supports and loading edge shall have the roughness less than 1.6 umRa specified in ISO 468.

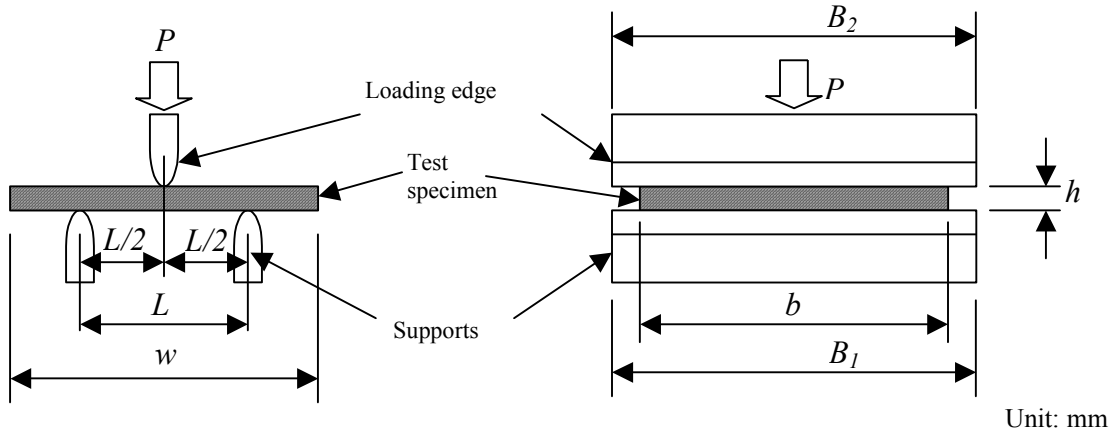


Figure 1
Position of Test Specimen at Start of Test

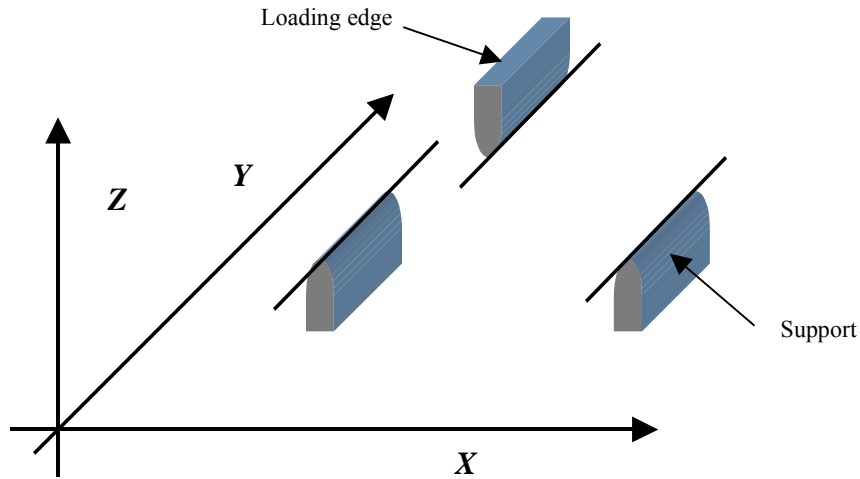


Figure 2
Arrangement of Supports and Loading Edge

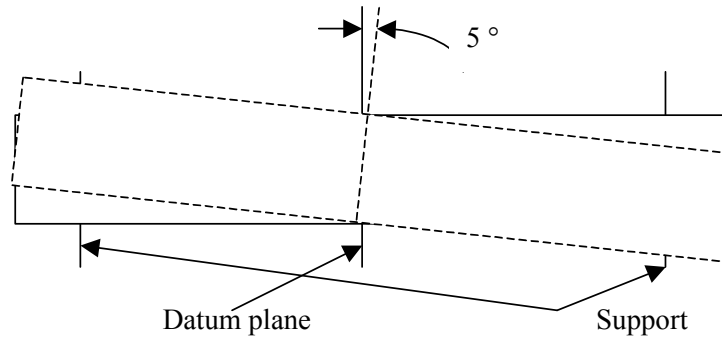


Figure 3
Setting Condition of Test Specimen

7 Test Specimens

7.1 The shape and dimensions of the test specimens shall be agreed between the interested parties.

7.2 Test specimen shall be Silicon Die.

7.3 At least 25 test specimens shall be pulled from a wafer.

8 Procedure

8.1 Measure the thickness h , width b , and length w . The accuracy of h and b shall be within $\pm 5\%$.

8.2 Adjust the span L in accordance with the following conditions,

at $h < 0.1 \text{ mm}$: $L \leq 2 \text{ mm}$ and $L \leq 50 h$

at $h \geq 0.1 \text{ mm}$: $2 \text{ mm} \leq L$ and $L \leq 20 h$

8.3 Place the test specimen on the two supports within $\pm 5^\circ$ of the deviation from datum plane (see Figure 3). The evaluated surface shall be applied the tensile force.

8.4 Set the test speed less than 5 mm/min in order to avoid the impact on test specimen and apply the force at midspan.

8.5 Record the force of the specimen at the break.

9 Calculation

9.1 *Flexural Stress* — Calculate the flexural stress parameters defined in Section 4 using the following equation:

$$\sigma_{JB} = \frac{3P_B L}{2bh^2} \quad (1)$$

Where:

σ_{JB} is the flexural stress at break in question (Mpa);

P_B is the applied force at break, in newtons (N);

L is the span, in millimeters (mm);

b is the width, in millimeters (mm), of the test specimen; and

h is the thickness, in millimeters (mm), of the test specimen.

NOTE 5: Equation (1) can only be used for test specimens with linear stress/strain behavior.

10 Test Report

10.1 The test report shall include the following information:

- the directions major axes of the specimens if test specimen has,
- the evaluated surface,
- the dimensions of the test specimens (thickness h , width b , length w),
- the nominal span length used,
- the number of specimens tested,
- the speed of testing,
- the individual test results, the flexural stress at break, and
- the date of the test.



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RELATED INFORMATION 1

ADDITIONAL INFORMATION FOR TESTING

NOTICE: This related information is not an official part of SEMI G86 and was derived from the work of the originating task force. This related information was approved for publication by full letter ballot procedures on January 10, 2003. It is provided for information purposes.

R1-1 Test Machine

R1-1.1 When the measurement of deflection is based on the movement of loading edge, the frame compliance of the test machine should be more than 40 kN/mm.

R1-1.2 The interval of data sampling, such as test force and movement of the loading edge, should be more than 500 times per second when the test machine records the data in digital format.

R1-2 Test Specimens

R1-2.1 The number of test specimen is 0.25 of Acceptance Quality Level, specified in ISO/DIS2859-1.2, Sampling procedures for inspection by attributes — Part 1: Sampling schemes indexed by acceptable quality level (AQL) for lot-by-lot inspection.

R1-2.2 Test specimens should be pulled from a wafer in every direction without leaning.

R1-2.3 When test results are compared, same test specimens size (span L and width b) should be used.

R1-2.4 When excessive film is attached to test specimens, equation (1) in Section 9.1 for stress on a silicon material may not be applied.

R1-3 Recording the Force

R1-3.1 Not only the force at the break points but also test force of the specimen under the test should be recorded. It is recommended that an automatic recording system is used.

R1-4 Test Report

R1-4.1 Test report should include the failure characteristics of the failed specimens.

R1-4.2 When possible, the test report should include failures which occur at any notable defect on the die.

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product data sheets, and other relevant literature, respecting any materials or equipment mentioned herein. These standards are subject to change without notice.

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SEMI INTERNATIONAL STANDARDS



PROCESS CHEMICALS

Semiconductor Equipment and Materials International



SEMI C1-0705

GUIDE FOR THE ANALYSIS OF LIQUID CHEMICALS

This guide was technically approved by the global Liquid Chemicals Committee. This edition was approved for publication by the global Audits and Reviews Subcommittee on April 7, 2005. It was available at www.semi.org in June 2005 and on CD-ROM in July 2005. Originally published in 1978; previously published July 2004.

1 Purpose

1.1 Liquid Chemicals are used extensively during the production of semiconductor devices; their purity has been shown to affect the device yield during the manufacturing processes.

1.2 Since there is a wide range of semiconductor devices being produced, different levels of purity have been established as standards and guidelines for each liquid chemical.

1.3 This guide describes the various analytical procedures for standards verification procedures required to meet a specific liquid chemical specification.

2 Scope

2.1 All liquid chemical standards consist of guidelines, specifications and validated analytical procedures. Suitable analytical procedures are defined in each standard for each grade of liquid chemical such that their sensitivity meets the requirements of each of the respective grades of liquid chemicals.

2.2 This guide provides an overview of each of the methods used for liquid chemical certification. Each grade of liquid chemicals (1–5) that have been validated analytically using the appropriate procedures described in this guide can be described as “meeting SEMI specifications.”

2.3 *Analytical Procedures* — The procedures used to establish a liquid chemical specification can be found in §7. The Analytical Procedures are listed in Table 1.

2.4 *Safety* — Because of the continuing evolution of safety precautions, it is impossible for this publication to provide definite statements related to the safe handling of individual liquid chemicals. The user is referred to product labels, product data sheets, government regulations, and other relevant literature.

NOTICE: This guide does not purport to address safety issues, if any, associated with its use. It is the responsibility of the users of this guide to establish appropriate safety and health practices and determine the applicability of regulatory or other limitations prior to use.

Table 1 Analytical Procedures

7.1	Acidity and Alkalinity
7.2	Assay by Wide Bore Column Gas Chromatography
7.3	Color
7.4	Residue after Evaporation
7.5	Residue after Ignition
7.6	Trace Arsenic (and Antimony) Determination
7.7	Use of a pH Meter
7.8	Ion Chromatography
7.9	Atomic Absorption Spectrometry (AAS)
7.10	Flame Emission Spectrometry
7.11	Graphite Furnace Atomic Absorption Spectrometry (GFAAS)
7.12	Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES)
7.13	Inductively Coupled Plasma Mass Spectrometry (ICP-MS)
7.14	Calibration and Measurement Method for Particles in Liquids

3 Referenced Standards and Documents

3.1 *ASTM Standards*¹

ASTM D5127 — Standard Guide for Ultra Pure Water Used in the Electronics and Semiconductor Industry

ASTM E203 — Standard Test Method for Water Using Volumetric Karl Fischer Titration

NOTICE: Unless otherwise indicated, all documents cited shall be the latest published versions.

4 Requirements

4.1 *Abbreviations and Signs* — In this guide abbreviations and signs used shall be restricted to those used in publications of the American Chemical Society (ACS)².

4.2 *Accuracy and Tolerances in Measurements* — In a test procedure, unless otherwise specified, the accuracy required in the amount of sample taken shall be understood to be within 2.0% of the stated amount, whether a weight or a volume. Where a dilution is performed to a stated volume or a stated volume of a solution is taken, that volume shall be within 2.0% of the stated amount.

4.3 *Acidity and Alkalinity* — A test for acidity or alkalinity, or both, of liquid chemicals may be specified. (See Acidity and Alkalinity in ¶7.1.)

4.4 *Analytical Blank* — Where a “blank” is specified to be run, unless otherwise stated, it shall be performed with the same quantities of the same reagents and in a manner identical to that followed with the sample portion under test, but with the material itself omitted.

4.5 *Apparatus* — Apparatus, unless otherwise qualified, shall be that commonly employed in the conduct of the relevant test.

4.6 *Atomic and Formula Weights* — Atomic weights (i.e. atomic masses) used shall be those of the latest publication of The International Union of Pure and Applied Chemistry (IUPAC)³. Formula weights shall be calculated from a given empirical (molecular) formula by addition of the stated multiples of the atomic weights. Unless otherwise stated, formula weights shall be rounded to 2 significant figures to the right of the decimal point (see Rounding of Numbers, ¶4.17).

4.7 *Comparison of Analytical Results with Specification Limits* — In the comparison of an analytical result for a test with the numerical limit associated with the specification, the result shall be rounded to the number of significant figures indicated for that limit. (See Rounding of Numbers, ¶4.17.) Consequently, a specification stated at 96% minimum will be met by a result as small as 95.5%, and that stated as 96.0% minimum will be met by a result as small as 95.95%. A specification of 0.1% maximum will be met by a result as large as 0.149%, and that of 0.10% maximum by a result as large as 0.105%. In those specifications given with no digit following the decimal point, such as 10 ng/mL maximum, a test result as large as 10.5 ng/mL will meet the specification. This use of a decimal point is made to clarify the difference between converting 0.01 mg/mL to 10 ng/mL and converting 0.010 mg/mL to 10. ng/mL.⁴

4.8 *Density* — The density of a liquid shall be determined for the compound in air at 25°C and be expressed in grams/milliliter (g/mL). Where the specific gravity, with respect to water, is determined, it shall be expressed as density by the use of tabular values for the density of water at the relevant temperature(s).

4.9 *Desiccator* — A desiccator shall imply a tightly closed container charged with a suitable desiccant (commonly calcium chloride or sulfate) that allows an atmosphere of low humidity to be maintained.

1 American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, Pennsylvania 19428-2959, USA. Telephone: 610.832.9585, Fax: 610.832.9555, Website: www.astm.org

2 <http://www.cas.org/ONLINE/standards.html>, American Chemical Society, 1155 Sixteenth Street, NW, Washington DC, 20036, Phone: 202-872-4600, Fax: 202-872-4615, Website: <http://www.chemistry.org>

3 The International Union of Pure and Applied Chemistry, PO Box 13757, Research Triangle Park, NC 27709-3757, USA, Website: <http://www.iupac.org>

4 H.A. Flachka, A.J. Barnard Jr., P.E. Sturrock. Quantitative Analytical Chemistry, Willard Grant Press, 2nd ed., 1980, p 4.

4.10 *Drying or Igniting to Constant Weight* — A statement “dried to a constant weight” or “ignited to a constant weight” shall imply that two separate weighings differ by no more than ± 0.4 mg (unless otherwise specified), where the second weighing follows a second drying (for one hour (unless otherwise stated) or ignition for 15 minutes (unless otherwise stated), respectively).

4.11 *Expression of Content and Concentration* — Unless otherwise stated, a specification limit and experimental results related to it shall be expressed in units of weight by weight. The concentration of a solution of a test reagent shall be expressed either in molarity or normality or as percent weight by volume. A redox normality value shall always be followed, parenthetically, by the relevant molarity. In the approximate dilution of reagents, a parenthetical expression of two numbers with an intervening “plus” sign shall imply that the relative volume of the stated reagent given by the first number shall be admixed with the relative volume of water given by the second number. Thus, “dilute” sulfuric acid (1 + 3) directs that one volume of reagent grade sulfuric acid be added to 3 volumes of water and the mixture stirred to form a uniform solution.

4.12 *Filtration* — A statement to “filter,” unless qualified, shall imply filtration through suitable filter paper until the filtrate is clear.

4.13 *Physical Properties* — Physical properties shall not usually be employed for specification purposes; for information, however, representative values for a particular liquid chemical, as supplied, may be included in the standard for that liquid chemical. Where relevant, physical properties shall be specified for 25°C.

4.14 *Reagent Chemicals* — Unless otherwise stated, reagents to be used in tests shall conform to the minimum standards of quality set forth in the current edition of Reagent Chemicals, published by the American Chemical Society⁵.

4.15 *Residue after Evaporation* — A “residue after evaporation” test may be used in the assessment of liquid chemicals. (See Determination of Residue after Evaporation, ¶7.4.)

4.16 *Residue after Ignition* — A “residue after ignition” test serves to assess the amount of nonvolatile inorganic matter present in a sample. (See Determination of Residue after Ignition, ¶7.5.)

4.17 *Rounding of Numbers* — The following rules for “rounding” of measured or calculated values shall be employed:

4.17.1 When the figure next beyond the last place to be retained is less than 5, leave unchanged the figure in the last place retained.

4.17.2 When the figure next beyond the last place to be retained is greater than 5, increase by 1 the figure in the last place retained.

4.17.3 When the figure next beyond the last place to be retained is 5 and there are no figures beyond this 5 or only zeroes, (a) increase by 1 the figure in the last place retained if it is odd, or leave the figure unchanged if it is even.

4.17.4 When the figure next beyond the last place to be retained is 5 and there are figures other than zeroes beyond this 5 increase by 1 the figure in the last place retained.

4.17.5 Obtain the rounded value in one step by direct rounding and not in two or more steps of successive roundings.

4.18 *Samples and Sampling* — For liquid chemicals provided in small containers, one (or more) should be freshly opened for testing, thereby reducing possibilities for contamination or change in composition (for example, by moisture pickup). Where the liquid chemical is provided in larger bulk quantities, one or more drums or other containers shall be sampled to avoid cross contamination, and the combined sample shall be placed in a labeled, well-cleaned container that shall be tightly closed and transferred expeditiously to the testing laboratory.

4.19 *Specifications and Specification Limits* — The specifications provided by this guide are intended to serve for liquid chemicals to be used in the manufacture and processing of semiconductors and advanced electronic devices and circuits. The specifications and the associated test procedures are based on the experience of suppliers and users and also on published studies relating to liquid chemicals of the required quality. The function of the specifications is to establish minimum standards of quality.

⁵ Available from American Chemical Society, 1155 Sixteenth Street, NW, Washington DC, 20036, Phone: 202-872-4600, Fax: 202-872-4615, Website: <http://www.chemistry.org>

4.19.1 Where feasible, a specification of content shall be expressed as a numerical limit in units of weight by weight. For a specification that cannot be assigned such a limit, the expression “To pass test” shall be used.

4.19.2 For a major component, the value (assay, purity, etc.) shall be expressed as a minimum permissible limit or range. For an impurity, the value shall be expressed as a maximum permissible limit.

4.19.3 A liquid chemical conforming to the specification will commonly contain either more of the major component than the minimum permissible limit or be within the specified range for the liquid chemicals and contain less of each impurity (or impurities) than the maximum permissible limit. In neither case shall the liquid chemicals be considered as of higher quality than that defined by the specification.

4.19.4 It is manifestly impossible in the specifications and procedures for a liquid chemical to consider every impurity or contaminant that might be present. For certain applications, it is recognized that more stringent or additional specifications and procedures might be required. The intent of these specifications and the associated procedures is, on one hand, to assure that a liquid chemical is suitable for the common uses to which it may be put in the manufacture and processing of semiconductor devices and, on the other hand, to be consistent with contemporary manufacturing processes for that liquid chemical.

4.20 *Specification Parameters* — The statement of specification parameters for a particular liquid chemical shall be, as far as practical.

4.20.1 Assay or other test assessing the component(s) of interest

4.20.2 Appearance or color

4.20.3 Acidity or alkalinity, or both, or pH range of solution

4.20.4 Residue after evaporation or ignition

4.20.5 Water content

4.20.6 Diverse general tests, arranged alphabetically

4.20.7 Tests for stated anions and ammonium shall be arranged alphabetically (ammonium, chloride, nitrate, phosphate, sulfate)

4.20.8 Tests for specific trace metals, arranged alphabetically by name. The 21 metals for grade specifications higher than Grade 2 are Aluminum, Antimony, Arsenic, Barium, Boron, Cadmium, Calcium, Chromium, Copper, Iron, Lead, Lithium, Magnesium, Manganese, Nickel, Potassium, Sodium, Tin, Titanium, Vanadium, and Zinc.

4.20.9 All major words in a specification parameter shall have their initial letters capitalized.

4.20.10 Following the statement of a specification parameter, where relevant, the species on which the calculation of the result is based shall be expressed parenthetically as the atomic symbol or empirical formula.

4.21 *Tared Containers* — Where the use of a tared container is specified, it shall be carried through operations identical to those used in the test procedures, including drying or ignition, or both, cooling in a desiccator, and weighing. When a new container is placed in service, special measures may be required to assure that it is brought to constant weight by the operations.

4.22 *Temperature* — Temperature values shall be expressed in degrees Celsius (°C).

4.23 *Water* — References to water in the testing procedures are understood to mean water that is of the appropriate grade for the intended purpose. Depending on the grade of liquid chemical being tested, water meeting the requirements for Types E-1, E-1.1, and E-1.2 water in ASTM Standard Guide D5127 may be used. In tests for nitrogen compounds, water should be “ammonia-free” or “nitrogen-free.” For some tests, freshly boiled water must be used in order to ensure freedom from material absorbed from the air such as ammonia, carbon dioxide, or oxygen.

4.24 *Water Content by Karl Fischer Titration* — The Karl Fischer method for the determination of water involves a titration with the so-called Karl Fischer reagent, consisting of iodine, sulfur dioxide, pyridine, and methanol. Details of this method are provided in the ASTM Standard Test Method E 203 and in Reagent Chemicals, published by the American Chemical Society

4.25 *Water and Steam Baths* — A water bath, unless otherwise stated, shall imply a bath of vigorously boiling water. A steam bath (at 1 atmosphere pressure), unless otherwise qualified, shall imply either exposure to flowing steam or to another source of heat at the temperature of flowing steam.

4.26 *Weights and Measures* — The weights and measures used shall be those of the International System (SI)⁶.

5 Reagent and Standard Solutions

5.1 The purity of reagents used in each analytical procedure should be concomitant with the grade of liquid chemical being analyzed.

5.2 *Ammonium Molybdate Reagent Solution* — Dissolve 50 g of ammonium molybdate, $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$, in 1 N sulfuric acid and dilute with 1 N sulfuric acid to 1000 mL.

5.3 *Ammonium Thiocyanate Reagent Solution* — Dissolve 150 g of ammonium thiocyanate, NH_4SCN , in water and dilute with water to 500 mL.

5.4 *Antimony Standard Solution* — Dissolve and dilute 0.275 g of potassium antimony $\text{KSbO}(\text{C}_4\text{H}_4\text{O}_6) \cdot 1/2\text{H}_2\text{O}$ with water to 1000 mL (1.0 mL = 0.1 mg of antimony (Sb)).

5.5 *Arsenic Stock and Working Solution* — See ¶7.6.3.1.

5.6 *Barium Chloride Solution* — Dissolve 60 g of barium chloride dihydrate, $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$, in water and dilute with water to 500 mL.

5.7 *Boron Standard Solution* — Dissolve 0.572 g of boric acid, H_3BO_3 , in water and dilute to 1000 mL with water. Then dilute 10 mL of this solution with water to 1000 mL (1.0 mL = 0.001 mg of boron (B)).

5.8 *“Bromine Water” Reagent Solution* — Add sufficient liquid bromine to water in a bottle so that undissolved bromine remains as a separate phase when the mixture is shaken.

5.9 *Brucine Sulfate Solution* — Dissolve 0.6 g of brucine sulfate, $(\text{C}_{23}\text{H}_{26}\text{N}_2\text{O}_4)_2 \cdot \text{H}_2\text{SO}_4 \cdot 7\text{H}_2\text{O}$, in dilute sulfuric acid (2 + 1), previously cooled to room temperature, and dilute to 1000 mL with the dilute acid. The sulfuric acid should be nitrate-free acid prepared as follows: Dilute the concentrated sulfuric acid (about 96% H_2SO_4) to about 80% H_2SO_4 by adding it to water, heat to dense fumes of sulfur trioxide, and cool. Repeat the dilution and fuming three or four times.

5.10 *Carminic Acid Reagent Solution* — Dissolve 0.05 g of carminic acid in 100 mL of sulfuric acid and shake until dissolution is complete.

5.11 *Chloride Standard Solution* — Dissolve 0.165 g of sodium chloride, NaCl , in water and dilute with water to 100 mL. Then dilute 10 mL of this solution with water to 1000 mL (1.0 mL = 0.010 mg of chloride (Cl) ion).

5.12 *Dimethylglyoxime Reagent Solution* — Dissolve 1.0 g of dimethylglyoxime in 100 mL of 95% EtOH (ethanol) reagent alcohol.

5.13 *Hydrogen Sulfide Water* — Immediately before use, saturate water with hydrogen sulfide gas.

5.14 *Hydroxylamine Hydrochloride Reagent Solution* — Dissolve 10 g of hydroxylamine hydrochloride, $\text{NH}_2\text{OH} \cdot \text{HCl}$, in water and dilute with water to 100 mL.

5.15 *Iron Standard Solution* — Dissolve 0.702 g of ferric ammonium sulfate hexahydrate, $\text{Fe}(\text{NH}_4)_2(\text{SO}_4)_2 \cdot 6\text{H}_2\text{O}$, in 10 mL of 10% sulfuric acid and dilute with water to 100 mL. Then to 10 mL of this solution, add 10 mL of 10% sulfuric acid and dilute with water to 1000 mL (1.0 mL = 0.010 mg of iron (Fe) ion).

⁶ http://www.bipm.fr/enus/3_SI/, Bureau International des Poids et Mesures, Pavillon de Breteuil, 92312 Sèvres cedex, France, Phone: +33 1 45 07 70 70, Fax: +33 1 45 34 20 21, Website: <http://www.bipm.fr/>

5.16 *Lead Standard Solution* — Dissolve 0.160 g of lead nitrate, $\text{Pb}(\text{NO}_3)_2$, in 100 mL of dilute nitric acid (1 + 99) and dilute to 1000 mL with water. Dilute 10 mL of this solution with water to 100 mL immediately before use (1.0 mL = 0.010 mg of lead (Pb) ion).

5.17 *Methyl Orange Indicator Solution* — Dissolve 0.1 g of methyl orange in 100 mL of water.

5.18 *Methyl Red Indicator Solution* — Dissolve 0.1 g of the hydrochloride salt form of methyl red in 100 mL of 95% EtOH (ethanol) reagent alcohol.

5.19 *p-(Methylamino)phenol Sulfate Reagent Solution* — Dissolve 0.2 g of p-(methylamino)phenol sulfate in 100 mL of water and add 20 g of sodium bisulfite. The performance of the resulting reagent solution in the determination of trace phosphate can be verified by the following test:

5.19.1 Place 25 mL of 0.5 N sulfuric acid and 1 mL of ammonium molybdate reagent solution in each of four test tubes. Add 1 mL of the reagent solution to each of the tubes. To the tubes add, respectively, zero, 0.005, 0.010, and 0.020 mg of phosphate ion (PO_4). After two hours at room temperature, an increasing intensity of blue color should be visible in the series.

5.20 *Nickel Standard Solution* — Dissolve 0.448 g of nickel sulfate hexahydrate, $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$, in water and dilute with water to 100 mL. Then dilute 10 mL of this solution with water to 1000 mL (1.0 mL = 0.010 mg of nickel (Ni) ion).

5.21 *Nitrate Standard Solution* — Dissolve 0.163 g of potassium nitrate, KNO_3 , in water and dilute with water to 100 mL. Then dilute 10 mL of this solution with water to 1000 mL (1.0 mL = 0.010 mg of nitrate (NO_3) ion).

5.22 *Nitrogen Standard Solution* — Dissolve 0.382 g of ammonium chloride, NH_4Cl , in water and dilute with water to 100 mL. Dilute 10 mL of this solution with water to 1000 mL (1.0 mL = 0.010 mg of nitrogen (N)).

5.23 *Phenolphthalein Indicator Solution* — Dissolve 1 g of phenolphthalein in 100 mL of 95% EtOH (ethanol) reagent alcohol.

5.24 *Phosphate Standard Solution* — Dissolve 0.143 g of monobasic potassium phosphate, KH_2PO_4 , in water and dilute to 100 mL with water. Then dilute 10 mL of this solution with water to 1000 mL (1.0 mL = 0.010 mg of phosphate (PO_4) ion).

5.25 *Platinum-Cobalt Stock Solution (APHA 500)* — (See ¶7.3.2.)

5.26 *Potassium Iodide Reagent Solution* — Dissolve 10 g of potassium iodide, KI, in 100 mL of water. Prepare immediately before use.

5.27 *Potassium Standard Solution* — Dissolve 0.191 g of potassium chloride, KCl, in water and dilute with water to 1000 mL. Dilute 100 mL of this solution with water to 1000 mL (1 mL = 0.010 mg of potassium (K) ion).

5.28 *Silver Nitrate Reagent Solution* — Dissolve 1.7 g of silver nitrate, AgNO_3 , in water and dilute with water to 100 mL.

5.29 *Sodium Carbonate Reagent Solution* — Dissolve 1.0 g of anhydrous sodium carbonate, Na_2CO_3 , in water and dilute with water to 100 mL.

5.30 *Sodium Arsenite Reagent Solution (5%)* — Dissolve 1.0 g of anhydrous sodium arsenite, NaAsO_2 , in water and dilute with water to 100 mL.

5.31 *Sodium Hydroxide Reagent Solution* — Dissolve 50 g of sodium hydroxide, NaOH, pellets in water and dilute with carbon dioxide-free water to 500 mL.

5.32 *Sodium Standard Solution* — Dissolve 0.254 g of sodium chloride, NaCl, in water and dilute with water to 1000 mL. Then dilute 100 mL of this solution with water to 1000 mL (1 mL = 0.010 mg of sodium (Na) ion).

5.33 *Stannous Chloride Reagent Solution* — Dissolve 20 g of stannous chloride dihydrate, $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$, in hydrochloric acid and dilute with hydrochloric acid to 100 mL.

5.34 *Starch Indicator Solution* — Mix 1 g of soluble starch for iodometry with a starch indicator and enough cold water to form a thin paste. Add 200 mL of boiling water and boil for 1 minute with stirring. Cool and transfer to a storage bottle that has been well rinsed with hot water.

5.35 *Sulfate Standard Solution* — Dissolve 0.148 g of anhydrous sodium sulfate, Na_2SO_4 , in water and dilute with water to 100 mL. Then dilute 10 mL of this solution with water to 1000 mL (1.0 mL = 0.010 mg of sulfate (SO_4) ion).

5.36 *Thymolphthalein Indicator Solution* — Dissolve 0.10 g of thymolphthalein in 100 mL of 95% EtOH (ethanol) reagent alcohol.

6 Method Validation

6.1 *Method Validation* — Analytical methods for trace metals, trace anions, moisture and assays require that performance parameters be assessed and compared to the standard and guideline requirements. The accuracy, precision of the measurement and interferences should be reviewed and reported with each method.

6.1.1 Accuracy for Trace Metals, Trace Anions, and Moisture

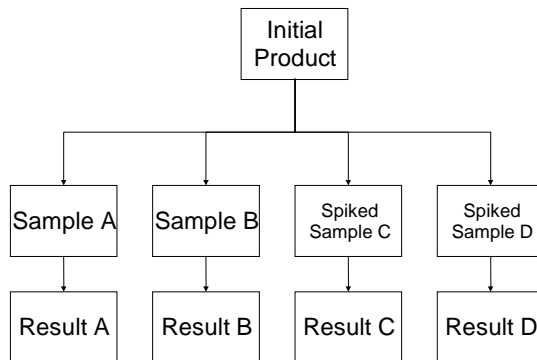
6.1.1.1 Method accuracy is a figure of merit that compares the data generated from the determined concentration of a sample to its “known” concentration. In some cases, a traceable standard may not be available and a “best available standard” may be substituted.

6.1.1.2 Determine the accuracy of a test method by utilizing a standard reference material as a sample and analyze it using the proposed test method. Report the test method accuracy as percent (%) bias:

$$\% \text{ bias} = \frac{X_{\text{"found"}} - X_{\text{"true value"}}}{X_{\text{"true value"}}} \times 100$$

6.1.1.3 If no suitable traceable standard exists for the “true value,” describe the procedures used to qualify the “best available standard.”

6.1.1.4 Spiked recovery determinations are used to make a limited assessment of accuracy. Method validation is based on the analysis of a minimum of two samples and a minimum of two spiked samples (Figure 1).



NOTE: Samples A, B, C, and D are originally from the same initial product.

Figure 2
Data Collection Structure

6.1.2 Measurement of Precision

6.1.2.1 Typically a repeatability and reproducibility study is done, where samples are analyzed over a period of multiple days.

6.1.2.2 Measures of method precision incorporate all applicable method variability sources (such as sample preparation, calibration, and instrumental performance) into their definitions. An assessment of method precision for trace metals, trace anions, and moisture is detailed in Tables 2 and 4.

Table 2 Data Reporting Requirements for Trace Metals, Trace Anions, and Moisture

Sample A	Result A
Sample B	Result B
Average	Average of Result A and B
Spike	The amount of spike equivalent to 50% of the specified value for trace impurity, and it should be added to the sample prior to any sample preparation (ex., sample dilution, evaporation, etc.)
Spike Sample C	Result C
Spike Sample D	Result D
Recovery 1	Result C – Average
% Recovery 1	(Recovery 1 / Spike) × (100)
Recovery 2	Result D – Average
% Recovery 2	(Recovery 2 / Spike) × (100)
Average % Recovery	(% Recovery 1 + % Recovery 2) / 2
% Recovery Range	Max. % Recovery - Min. % Recovery
Result Standard Deviation	When applicable, report the Std. Dev. of multiple measurements for each Result
Relative Standard Deviation	When applicable, report the Relative Std. Dev. of multiple measurements for each Result

6.1.2.3 Measurement Repeatability targets how well measurements can be repeated and should be reported as percent (%) relative standard deviation (Tables 2 and 4).

6.1.3 Interference Checks

6.1.3.1 List all interferences that were examined during the course of method validation. Also note any known potential interferences.

6.1.4 Data Reporting Requirements

6.1.4.1 Table 2 along with Figure 1 illustrates the minimal data structure and analysis required to support method validation for trace metals, trace anions, and moisture. Table 3 is used for assays.

Table 3 Data Reporting Requirements for Assays

Specification Width	W = upper spec. limit – lower spec. limit If no upper spec. limit is stated for an assay of an ultrapure product, use 100%.
Pooled Standard Deviation	$s_p = \sqrt{s_p^2} = \sqrt{\frac{\sum_{i=1}^N (n_i - 1)s_i^2}{\sum_{i=1}^N (n_i - 1)}}$ <p>where $N \geq 1$ groups of current or historical $n_i \geq 2$ assays taken on the i^{th} sample have their standard deviations pooled and s_i^2 is the standard deviation of the data in the i^{th} group squared.</p>
Degrees of Freedom	$df = \sum_{i=1}^N (n_i - 1)$ <p>where degrees of freedom ≥ 4</p>

90% Upper Confidence Limit	$90\% \text{ UCL} = t_{df, 0.10} \frac{s_p}{\sqrt{n}}$ <p>where n is the total number of assay measurements taken on the sample in question and t is a t-statistic with the associated degrees of freedom and 10% of the distribution to its right</p>
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6.1.4.2 In cases for trace metals, trace anions, and moisture where more samples are measured, statistical quantities analogous to those in Table 2 should be constructed. For methods where automatic consecutive measurements can be obtained from the instrument, Results A, B, C, and D should be the mean of five or more instrumental measurements.

6.1.5 *Summary of Criteria for Success* — The success of the criteria for each figure of merit depends on the specification, the matrix of the analyte, and levels of detection required. For trace metals, trace anions and moisture, method success criteria are found in Table 4. For assays the 90% upper confidence limit can not exceed 30% of the specification's width.

Table 4 Method Success Criteria for Trace Metals, Trace Anions and Moisture

<i>Figure of Merit</i>	<i>Criteria for Successful Method</i>
Accuracy	
Spiked Recovery Study	75-125% Spiked Recovery for each spiked sample.
Method Precision	
Spiked Recovery Study	% Recovery Range less than or equal to 35%.
Measurement Precision	Maximum RSD = 20% of the concentration of each spiked sample where: $\text{RSD} = \frac{\text{Std.Dev. Result}}{\text{Conc. of Spiked Sample}} \times 100$
Method Detection Limit	As per SEMI C10: <i>Guide for Determination of Method Detection Limits</i> ; Method Detection Limits cannot exceed the specification.
Interference Checks	If interferences were examined or potential interferences exist, list them.

6.1.6 *Applicability to Existing Specifications* — The current implementation of Method Validation only applies to new or revised specifications. Any prior method validated by SEMI to support a given specification still remains applicable to that specification.

6.1.7 *Example: Trace Metals, Trace Anions and Moisture* — Suppose that repeat measurements from four samples; A, B, C and D were available as follows:

Sample A: 0.19, 0.21, 0.25, 0.15, 0.23

Sample B: 0.31, 0.21, 0.17, 0.22, 0.26

Spiked Sample C: 0.83, 0.62, 0.76, 0.79, 0.80

Spiked Sample D: 0.77, 0.87, 0.69, 0.83, 0.84

NOTE 2: Table 5 illustrates the calculations required as per Table 2.

Table 5 Data Reporting – Example 6.1.7

Sample A	$(0.19 + 0.21 + 0.25 + 0.15 + 0.23) / 5$ = 0.206
Sample B	$(0.31 + 0.21 + 0.17 + 0.22 + 0.26) / 5$ = 0.234
Average	$(0.206 + 0.234) / 2 = 0.220$
Spike	The spike level is to be $\frac{1}{2}$ the relevant specification. If the specification is 1 ppb, then the spike would be 0.5 ppb.
Spike Sample C	$(0.83+0.62+0.76+0.79+0.80) / 5$ = 0.760
Spike Sample D	$(0.77+0.87+0.69+0.83+0.84) / 5$ = 0.800
Recovery 1	$(0.760 - 0.220) = 0.540$
% Recovery 1	$(0.540 / 0.5) = 108$
Recovery 2	$(0.800 - 0.220) = 0.580$
% Recovery 2	$(0.580 / 0.5) = 116$
Average % Recovery	$(108 + 116) / 2 = 112$
% Recovery Range	$(116 - 108) = 8$
Result Standard Deviation	Since repeat measurements were available in this example, report the sample standard deviations. Sample A: 0.038 Sample B: 0.053 Sample C: 0.082 Sample D: 0.071
Relative Standard Deviation (as a %)	Since repeat measurements were available in this example, report the relative sample standard deviations. Samp. A: $100 \times (0.038 / 0.220) = 17.5$ Samp. B: $100 \times (0.053 / 0.220) = 24.2$ Samp. C: $100 \times (0.082 / 0.720) = 11.4$ Samp. D: $100 \times (0.071 / 0.720) = 9.9$ Note use of the average of 0.220 and the sum of the average + spike (0.720) above.

NOTE 3: This example provided the data for the first three success criteria found in Table 4 as well as providing other required summary statistics. Table 6 summarizes performance relative to the success criteria.

Table 6 Method Success Criteria for Trace Metals, Trace Anions, and Moisture – Example 6.1.7

<i>Figure of Merit</i>	<i>Criteria for Successful Method</i>
Accuracy	Recovery 1 (108%) as well as Recovery 2 (116%) are in the required 75–125% Spiked Recovery range.
Method Precision	The % Recovery Range was 8% and meets the requirement of not exceeding 35%.
Measurement Precision	The maximum RSD for the spiked recoveries is 11.4% which is less than the maximum allowable 20%. Note that the RSDs calculated for Samples A and B are not relevant for this requirement.

<i>Figure of Merit</i>	<i>Criteria for Successful Method</i>
Method Detection Limit	Not illustrated in this example, an example is provided with SEMI C10. The Method Detection Limit must be less than or equal to the specification.
Interference Checks	List as necessary.

6.1.8 *Example: Assay With Stated Upper and Lower Specification Limits* — Suppose that the liquid chemical in question has a target specification of 28% with a lower specification limit of 27% and an upper specification limit of 29%. The specification width is 2% = 29% – 27%. Suppose the following data was available all as percentages:

Historical Sample A: 28.9, 29.1, 29.9

Historical Sample B: 27.8, 27.6, 27.6, 27.7

Current Sample: 28.1

For purposes of calculating the degrees of freedom and the pooled standard deviation as per Table 7:

$$n_1 = 3, s_1^2 = 0.280, n_2 = 4, s_2^2 = 0.009,$$

Where

$$s_1^2 = 0.280 = (0.52915)^2 = [\text{std. dev. (28.9, 29.1, 29.9)}]^2$$

$$s_2^2 = 0.009 = [\text{std. dev. (27.8, 27.6, 27.6, 27.7)}]^2$$

The degrees of freedom = 5 = (3-1) + (4-1) exceeds the minimum requirement of 4 and the pooled standard

deviation is calculated as:

$$s_p = \sqrt{\frac{\sum_{i=1}^N (n_i - 1) s_i^2}{\sum_{i=1}^N (n_i - 1)}} = \sqrt{\frac{2(0.280) + 3(0.009)}{2 + 3}}$$

$$s_p = \sqrt{0.1174} = 0.343$$

The 90% UCL is calculated as: 90% UCL = $t_{df, 0.10} \frac{s_p}{\sqrt{n}} = 1.476 \frac{0.343}{\sqrt{1}} = 0.506$

Where $n = 1$ = number of current meas. taken on sample and the t statistic is from Table 7. Since the 90% UCL / spec. width = 0.506/2 = 25.3% < 30% method validation is established.

Table 7 t-statistics for Assay

<i>Degrees of Freedom</i>	<i>t-statistic</i>
4	1.533
5	1.476
6	1.440
7	1.415
8	1.397
9	1.383
10	1.372
11	1.363
12	1.356

Degrees of Freedom	t-statistic
13	1.350
14	1.345
15	1.341
16	1.337
17	1.333
18	1.330
19	1.328
20	1.325

6.1.9 *Example: Assay With Only a Stated Lower Specification Limit* — Suppose that the liquid chemical in question has a minimum purity level of 99.5%; i.e., a lower specification limit of 99.5% with an unstated upper specification limit of 100%. The specification width is 0.5% = 100% - 99.5%. Suppose the following data was available all as percentages:

Historical Sample A: 99.65, 99.63, 99.60 99.63

Current Sample: 99.58, 99.62

For purposes of calculating the degrees of freedom and the pooled standard deviation as per Table 3:

$$n_1 = 4, s_1^2 = 0.000425, n_2 = 2, s_2^2 = 0.0008$$

The degrees of freedom = 4 = (4-1) + (2-1) meets the minimum requirement of 4 and the pooled standard

deviation is calculated as:

$$s_p = \sqrt{\frac{\sum_{i=1}^N (n_i - 1)s_i^2}{\sum_{i=1}^N (n_i - 1)}} = \sqrt{\frac{3(0.000425) + 1(0.0008)}{3 + 1}}$$

$$s_p = 0.023$$

The 90% UCL is calculated as: $90\% \text{ UCL} = t_{df, 0.10} \frac{s_p}{\sqrt{n}} = 1.533 \frac{0.023}{\sqrt{2}} = 0.0249$

Where $n = 2$ = number of current meas. taken on the sample and the t statistic is from Table 7. Since the 90% UCL / spec. width = 0.0249/0.5 = 5.0% < 30% method validation is established.

7 Analytical Procedures

7.1 Acidity and Alkalinity

7.1.1 In the acidity test, usually a stated volume of water (commonly 25 mL) is shaken in a glass-stoppered flask with a stated volume of sample (commonly 10 mL). Then 0.1 mL of phenolphthalein indicator solution is added and 0.01 N sodium hydroxide until a slight pink color persists after shaking for 30 seconds. Next, a stated amount of sample is added and mixed, and a titration is performed with 0.01 N sodium hydroxide until the pink color is reproduced; the volume of titrant required is recorded.

7.1.2 In the alkalinity test, a stated amount of sample is mixed with a stated volume of water (commonly 25 mL). Now 0.05 mL of methyl red indicator solution is added and the mixture is titrated with 0.01 N hydrochloric acid until a slight pink color is reached; the volume of titrant required is recorded.

7.1.3 The result of such tests has often been expressed qualitatively as “passes test” or quantitatively as the content of an acid (or base) known or assumed to be present. In this guide, the maximum specification limit shall be expressed in micro equivalents of acid (or base) per gram of sample (µeq/g). The maximum allowed volume of titrant in a test procedure shall correspond to that specification limit.

7.2 Assay by Wide Bore Column Gas Chromatography — The theory of gas chromatography is given in many documents, reviews, etc. The purpose here is to delineate some practical aspects for the assessment of volatile organic reagents by gas chromatography. A complete detailed procedure for the gas chromatographic assay is not given since available instruments vary from laboratory to laboratory. However, the use of wide bore capillary columns is recommended to replace conventional packed columns. Capillary columns, constructed of fused silica onto which is bonded a liquid phase, have greater resolving power and liquid chemical inertness.

7.2.1 Equipment — A gas chromatograph equipped with capillary wide bore column adaptors of isothermal and multi-step linear and temperature-programmed operation is recommended.

7.2.2 Sample — Direct flash vaporization or on-column injection of the sample with standard gauge needles can be used with wide bore columns. On column injection can minimize sample degradation while increasing the reproducibility of results. On column injection is ideal to use with very volatile materials such as the reagents assayed in this guide.

7.2.3 Columns — Many types of columns exist and can be used. Until recently, packed columns in which a liquid phase is coated on a porous solid support were extensively used for assay of reagents. The introduction of wide bore capillary columns having an i.d. of typically 530 micrometers and a thick film of liquid phase from 1 to 5 micrometers allows a laboratory to use a packed column instrument and conditions, while gaining the advantages of capillary technology. Columns can be used either in the high resolution capillary mode (low carrier gas flow rates) to achieve optimum resolution of sample components, or at higher flow rates (20–30 mL/min) where they will perform packed column-like separations in a shorter time. Packed columns require a multitude of stationary phases to accomplish typical separations performed in an analytical laboratory. The increased length of capillary columns allows for better separation of components so that three columns of low (Type 1, Methyl Silicone), moderate (Type 2, Mixed Cyano, Phenyl, Methyl Silicone), and high (Type 3, Carbowax) polarity can handle the majority of analytical requirements. Columns, which have been found acceptable for assay of the reagents in this manual, are listed in Table 8.

Table 8 Columns for Wide Bore Column Gas Chromatography

<i>Reagent</i>	<i>Recommended^{#1} Column</i>	<i>Alternate^{#2} Column</i>
Acetone	III	II, I
n-Butyl Acetate	I	II
Dichloromethane	I	II
Methanol	I	
Methyl Ethyl Ketone	I	
2-Propanol	I	III
Tetrachloroethylene	I	
Toluene	I	III
Trichloroethylene	I	
Trichlorotrifluoroethane	I	
Hexamethyldisilazane ^{#3}	I	
1-Methyl-2-Pyrrolidone	I	
1,1,1 Trichloroethane	I	

Type I Column — Methyl Silicon Bonded Phase

Type II Column — Mixed Cyano, Phenyl, Methyl Silicon Bonded Phase

Type III Column — Carbowax Bonded Phase

#1 Satisfactory for both assay and SEMI-specified impurity determinations.

#2 Column will separate all SEMI-specified impurities from assay component. Elution order will differ from typical given in individual reagent procedures.

#3 Presilanized column recommended.

7.2.4 Conditions — Appropriate conditions will vary from one gas chromatographic system to another. Conditions, which have been found acceptable, are listed under each individual standard. When using packed column in the isothermal mode, the temperature of the column is usually maintained at 10° to 20°C below the boiling point of the

reagent. The efficient mass transfer associated with wide bore capillary columns may necessitate initial column temperatures to be more than 20°C below the boiling point of the reagent of interest. Temperature programming may be used to improve the shape of eluting peaks and to shorten analysis times. The remaining two heated zones (injector and detector) should be maintained at as low a temperature as possible to lessen thermal decomposition effects. Typically, for the injector this is 50°C above the boiling point of the material of interest; the detector is held 25°C above the highest temperature reached by the column, but never less than 100°C. The split or splitless mode of injection can be used depending on the chromatographic equipment available. The use of a wide bore capillary column simplifies conversion of a packed column to a capillary instrument.

7.2.5 Detector — Use of a thermal conductivity detector minimizes the need to correct for detector response to differences in liquid chemical composition and is usually sufficient for determining reagent assay. To qualify trace impurities, the more sensitive flame ionization detector is recommended. The detector output, after amplification, is used to produce the chromatogram, a plot of component quantity versus elution time. Modern systems digitize the analog output, allowing direct printout of peak areas and retention times.

7.2.6 Calculation of Results — Results from a chromatogram, used to determine the assay of a reagent, are expressed in area percent normalized to 100%. A response factor correction for each component is required for the most accurate results, especially when the sample components differ markedly in their detector response. An internal standard reduces error due to variations in injection quantities, column, and detector conditions. The use of control charts, to aid the analyst in visualizing chromatographic variability, is suggested. To verify that assay results are valid, use of a system suitability test is recommended.

7.3 Color

7.3.1 Color (APHA) — For the rapid assessment of “colorless” liquids for contamination by colored materials, visual comparison with platinum-cobalt solution standards is appropriate, following the methodology of the American Public Health Association (APHA)⁷ established for the evaluation of the color of water.

7.3.2 Preparation of Platinum-Cobalt Stock Solution (APHA No. 500) — Dissolve 1.246 g of reagent grade potassium chloroplatinate, K_2PtCl_6 , and 1.000 g of cobalt chloride hexahydrate, $CoCl_2 \cdot 6H_2O$, in water. Add 100 mL of reagent grade hydrochloric acid (~37%) and dilute with water to exactly 1,000 mL. Store in a tightly closed bottle.

7.3.3 General Procedure for “Color (APHA)” Test — For the working standard, dilute one or more suitable aliquots of the Platinum-Cobalt Stock Solution (APHA No. 500) with water to 100 mL. In 100 mL Nessler tubes, compare the working standard with an equal volume of the liquid under assessment. View each tube vertically over a white background. The color of the liquid should not exceed the maximum APHA standard value allowed by the specification for that liquid. On the dilution to 100 mL, aliquots of the stock standard of 1, 2, 3 . . . mL correspond to APHA 5, 10, 15, etc.

7.4 Determination of Residue after Evaporation

7.4.1 Introduction — The residue after evaporation is defined as the relative amount of residue remaining after evaporation of a sample of solvent followed by heating of the residue at 105°C for 30 minutes. Drying to constant weight is not utilized since protracted heating may lead to slow volatilization of high-boiling matter.

7.4.2 For the determination of the residue after evaporation at the level of 5 parts per million ($\mu\text{g/g}$) or below, determinate errors must be kept extremely small and random errors should be minimized. In the procedure given below, the positive error due to exposure to airborne particulate matter is reduced by the use of the closed system afforded by the Thiers assembly. Possible negative errors associated with losses caused by transfer of a concentrated sample or the residue is avoided by effecting the evaporation and weighing in a single dish. The determinate and random errors associated with the weighing operations are reduced by using an aluminum dish for most solvents, weighing on a semi-microbalance, eliminating any static electricity, and conducting all heating and cooling steps in a reproducible manner.

7.4.3 Equipment — The Thiers assembly is fashioned from a crystallizing dish. (See Figure 2.) The aluminum dishes of 250 mL capacity are prepared by cutting out a 30 cm circle of aluminum foil, rinsing it with the solvent,

7 American Public Health Association, 800 I Street, NW, Washington, DC 20001, Phone: (202) 777-2742, Fax: (202) 777-2534, Website: <http://www.apha.org>

and, using gloves, pressing it around the base of an 800 mL beaker. The balance is a semi-micro type, and the static electricity eliminator may be obtained from supply houses.

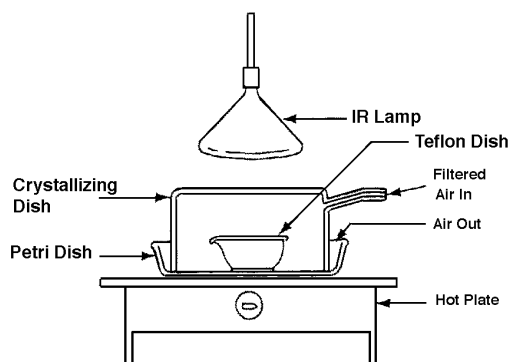


Figure 3
Thiers Assembly

7.4.4 Procedure

7.4.4.1 Rinse the 250 mL aluminum dish (or platinum dish in the case of halogenated solvents) with a portion of the sample. Heat the dish at 105°C in an oven for 30 min and cool for 30 min (1 hr for platinum) in a desiccator charged with calcium chloride and placed near the balance. Discharge static electricity using the eliminator and weigh the dish on a semi-microbalance to the nearest 0.01 mg.

7.4.4.2 Start the flow of filtered nitrogen gas through the side arm of the Thiers assembly and place the dish inside. Raise the cover briefly to add the specified amount of solvent from a clean, graduated cylinder. Adjust the nitrogen stream so that the surface of the solvent is barely rippled. Also, adjust the heat lamp and hot plate so that the evaporation rate does not exceed 8 mL/min and that no sample condenses on the top inner surface of the assembly.

7.4.4.3 When all of the sample has evaporated, heat the dish for 30 min in an oven at 105°C. Cool as given above, discharge the static electricity, and reweigh. Calculate the residue as mg/mL.

$$\frac{[\text{wt of dish plus residue (g)} - \text{wt of dish (g)}] \times 10^6}{\text{sample wt (g)}}$$

For best results, room temperature should be kept constant to $\pm 5^\circ\text{C}$.

7.5 *Residue after Ignition* — A “residue after ignition” test serves to assess the amount of nonvolatile inorganic matter present in a sample. Unless otherwise stated, the ignition shall be performed at $800^\circ \pm 25^\circ\text{C}$ for 15 minutes. Where the addition of sulfuric acid is specified, the result corresponds to what is often termed “sulfated ash.” The calculation of the result, expressed in parts per million ($\mu\text{g/g}$), takes the form:

$$\text{ppm Residue after Ignition} = \frac{\text{Net weight of residue} \times 10^6}{\text{Weight of sample}}$$

7.6 Trace Arsenic (and Antimony) Determination

7.6.1 *Introduction* — This photometric procedure is based upon the color reaction of silver diethyldithiocarbamate with arsine (and stibine). It provides for measurement of the absorbance of the sample and standards with subsequent calculation of the arsenic and antimony content of the sample, expressed as arsenic. The procedure given below differs from conventional ones by the omission of potassium iodide and stannous chloride. By this omission, antimony is evolved as stibine and reacts with the silver diethyldithiocarbamate solution. The color development is consequently due to both arsenic and antimony. The test sensitivity for arsenic is greater than for antimony.

7.6.2 *Apparatus* — The apparatus (see Figure 3) consists of a 125 mL arsine generator (a) fitted with a scrubber unit (c) and an absorber tube (e), with a 24/40 standard-taper joint (b) and a ground glass ball-and-socket joint (d), secured with a No. 12 clamp, connecting the units. In order to insure a more uniform rate of flow of gas into the absorbing solution, various modifications may be employed, such as the use of a fritted-glass disk sealed into the tapered end of the absorption tube.

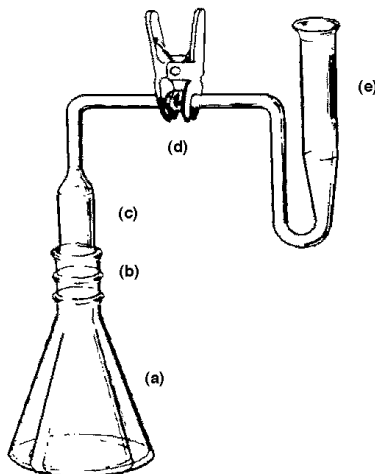


Figure 4
Arsenic Test Apparatus

7.6.3 Reagents

7.6.3.1 *Arsenic Stock Solution* — (1.00 mg/mL of arsenic) — Transfer 0.66 g of arsenic trioxide to a 500 mL volumetric flask and dissolve with 5 mL of 50% sodium hydroxide solution. Neutralize with 5 N sulfuric acid, add an excess of 5 mL, dilute to the mark with water, and mix thoroughly.

7.6.3.2 *Arsenic Working Solution* — (1.0 µg/mL of arsenic) — Transfer exactly 0.5 mL of the arsenic stock solution to a 500 mL volumetric flask, and 5 mL of 5 N sulfuric acid, dilute to the mark with water, and mix thoroughly. Prepare immediately before use.

7.6.3.3 *Lead Acetate Solution* — Dissolve 10 g of lead acetate trihydrate in 100 mL of water.

7.6.3.4 *Silver Diethyldithiocarbamate Solution* — Dissolve 1 g of diethyldithiocarbamic acid, silver salt, in 200 mL of pyridine and filter the solution. (The pyridine is best purified by passage through an alumina column.) Store this solution in a light-resistant container and use within 1 month. Silver diethyldithiocarbamate as a dry solid is yellow in color. Discard material that is markedly off color or develops a strong odor; alternatively, recrystallize from 1:1 pyridine-water and dry in vacuo at room temperature.

7.6.4 *Sample Solution Preparation* — Prepare the sample solution as specified in the procedures for the individual liquid chemical.

7.6.5 Procedure

7.6.5.1 Treat the sample as described in the procedure for the individual chemical and transfer the resulting solution to the generator flask. Swirl the flask, add 5 mL of 20% sulfuric acid, and allow to stand at room temperature for 15 minutes.

7.6.5.2 Pack the scrubber tube (c) with two pledgets of lead acetate-impregnated glass wool previously moistened with lead acetate solution freed from excess solution by squeezing and dried in a vacuum. Allow a small space between the two pledgets. Place 3.0 mL of silver diethyldithiocarbamate solution in the absorber tube (e), add 3 g of zinc (granular) to the generator flask (a), and immediately connect the scrubber-absorber assembly to the flask.

7.6.5.3 Place the generator flask in a water bath maintained at $25^{\circ} \pm 3^{\circ}\text{C}$ and swirl the flask gently at 10 minute intervals. (The addition of a small volume of 2-propanol to the generator flask may make the gas evolution more uniform.) After 45 minutes, disconnect the tubing from the generator flask, transfer the silver diethyldithiocarbamate solution to a 1 cm photometer cell, and measure the absorbance at 525 nm versus the water blank.

7.6.5.4 Carry a water blank and standard through the procedure, using the identical quantities of the same reagents. The standard consists of a volume of the arsenic working solution corresponding to the amount of arsenic stated in the procedure for the individual liquid chemicals. Any red color in the silver diethyldithiocarbamate solution from the sample preparation should be no greater than that obtained with the standard.

7.7 *Use of a pH Meter* — The principles of pH measurement are given in documents and instrument manuals. The purpose here is to describe in general terms how the technique is used with the procedures for individual liquid chemicals, either to assure that a “pH of Solution” specification is met or to adjust the acidity or alkalinity of a solution as required in the conduct of a procedure.

7.7.1 *Equipment* — The pH meter should be capable of reading to 0.02 pH units and should best be equipped for temperature compensation. The electrodes should be calomel and glass and should be kept in appropriate solutions when not in use, following the supplier’s recommendations.

7.7.2 *Standardization* — At time of use, the pH meter and electrodes should be standardized against two pH standard buffers that preferably should bracket the expected pH of the solution to be measured. The meter with the electrodes immersed in the first buffer should be adjusted to the stated value for that buffer. The electrodes should then be rinsed, and the meter, with the electrodes immersed in the second buffer, should be adjusted to the stated value for that buffer. This adjustment procedure should be repeated until two consecutive measurements for one of the buffers gives the same value within ± 0.02 pH units. Where a temperature is specified for an individual liquid chemical, either as part of the specification parameter or the relevant procedure, that temperature should be maintained in the standardization with the corresponding assigned pH values for the pH standard buffers being employed. During measurements, each buffer should be magnetically stirred at the same constant rate.

7.7.3 *Quantitation* — For the measurement of the pH of a solution, the meter and electrodes should be standardized as given above. The electrodes should then be rinsed with water and immersed in the solution (held at the specified temperature), and the pH read once the reading has stabilized. The solution should be magnetically stirred at the same rate as used in the standardization. If the reading does not stabilize within 1 minute, the cause should be found and eliminated. Any water used to dilute a sample for the “pH of Solution” test should be carbon dioxide-free.

7.8 *Determination of Various Ions by Ion Chromatography* — The theory of ion chromatography is given in many documents, reviews, etc. The purpose here is to delineate some practical aspects for the determination of various ions by Ion Chromatography. A complete detailed procedure for the ion chromatographic determination is not given since available instruments vary from laboratory to laboratory.

7.8.1 *Equipment* — An ion chromatograph equipped with ion suppression is recommended. Single column ion chromatography can also be used. The choice of reagents and columns is determined by the ions of interest.

7.8.2 *Sample* — Sample preparation varies depending upon the matrix effects of the liquid chemicals being analyzed. Standard sample preparation techniques that may be used are (this list is not all inclusive):

7.8.2.1 Evaporation of the reagent to remove the matrix and then diluting the residual with the appropriate grade of 18.2 Mohm-cm water that meets ASTM Standard Guide D5127.

7.8.2.2 Diluting the reagent with the appropriate grade of 18.2 Mohm-cm water that meets ASTM Standard Guide D5127 to lessen the matrix effects.

7.8.2.3 Direct injection of the reagent.

7.8.3 *Columns* — Many types of columns exist and can be used. Typical selection criteria for columns consist of ion or ions of interest, the matrix of the reagent and eluent to be used.

7.8.4 *Conditions* — Appropriate conditions will vary from one ion chromatographic system to another. Analytical conditions using ion suppression, which have been found acceptable using ion suppression, are listed under each individual standard. The two broad classifications of ions are anions and cations. A typical eluent used in the analysis of anions by ion suppression is a sodium carbonate and sodium bicarbonate in a solution with the appropriate grade of 18.2 Mohm-cm water that meets ASTM Standard Guide D5127. A typical eluent used in the

analysis of cations is a dilute solution of hydrochloric acid in a solution with the appropriate grade of 18.2 Mohm-cm water that meets ASTM Standard Guide D5127.

7.8.5 Eluent concentration, eluent flow rate, and system pressure are variables, which affect the chromatography. Each must be determined for the chromatographic system being used.

7.8.6 *Detector* — A conductivity detector is used to monitor the changes in the eluent conductivity due to ions in the sample. An ultraviolet absorbance detector can be used for detection of some ions. The detector output, after amplification, is used to produce the chromatogram, a plot of component quantity versus elution time. Modern systems digitize the analog output allowing direct printout of peak heights, peak areas, and retention times.

7.8.7 Calculation of Results

$$\text{Ion Concentration} = \frac{(\text{Pk. Ht. } S_i - \text{Pk. Ht. } B_i)(\text{Conc. of Std. } i)}{\text{Pk. Ht. Std. } i - \text{Pk. Ht. } B_i} \times \text{Dilution Factor}$$

Pk. Ht. S_i = Peak Height of Sample Ion

Pk. Ht. B_i = Peak Height of Blank Ion

Pk. Ht. Std_i = Peak Height of Standard Ion

Conc. of Std_i = Concentration of Standard Ion

Peak area may be used instead of peak height. Calibration linearity must be established before use.

7.9 *Determination of Trace Elements by Atomic Absorption Spectrometry (AAS)* — The purpose here is to describe in general terms how the technique should be used when specified in the procedures for an individual liquid chemical to assure that the specifications for one or more stated elements are met. No general procedure is given since operating details will vary with instrument design; consequently, the manufacturer's manual should be followed.

7.9.1 *Equipment* — The source is a hollow cathode lamp, or equivalent, for the relevant element. The burner is slotted and will vary in design and path length according to the gas(es) to be used. The monochromator should have a resolution of at least 0.1 nm. The signal from the detector is read from a meter, digital display, recorder tracing or custom software. A background correction system should be available.

7.9.2 *Flame and Burner Conditions* — The flame gases to be used are specified in the procedure for the individual liquid chemical. The burner position and gas flow rates should be optimized for a given determination.

7.9.3 *Solution Conditions* — The solutions are prepared as given in the procedures for the individual liquid chemical.

7.9.4 *Working Standard Solutions* — Working standard solutions should be prepared on the day of use by dilution of a stock standard solution.

7.9.5 *Quantitation* — Commonly, the method of standard addition is employed. It is applicable at low concentrations where a linear relationship exists between signal and concentration. By this technique the standards have a matrix similar to the sample. It is essential that the sample solution, along with the additions, has absorbance in the range for which linearity is expected: routinely, 0.1 to 0.5 absorbance unit. The use of standard additions does not eliminate interferences, but assures that the element of interest behaves similarly in the sample preparation and those with additions. Background correction is usually required, and additives may be called for (see below).

7.9.5.1 For specific tests, the signals are measured for five solution preparations: the reagent blank (which may be only water), the sample solution, and three made by standard additions to the sample solution. The signal for the reagent blank preparation is subtracted from the signals for each of the four other preparations. These four signals are used graphically or mathematically to determine the content of the element in the sample. Where several elements are to be determined, they are added simultaneously to the third, fourth, and fifth preparations. Commonly, the additions correspond to one half the specification limit, equal to the limit, and twice the limit.

7.9.5.2 A calibration line obtained for aqueous standards can be used if its slope and the slope of the standard addition line are identical. In that event, liquid chemical (matrix) effects are negligible.

7.9.6 Background Correction — Where spurious absorption or other background correction is required, details are given in the procedure for an individual liquid chemical. For an element with the stated resonance line in the ultraviolet region (<350 nm), the correction should be performed with a deuterium discharge lamp. For an element with the stated line in the visible region (>350 nm), the adjacent line technique should be employed. In this method, the background absorption is determined at a wavelength adjacent, ± 5 nm, to the line being used for the element of interest; that is, it is measured at a wavelength where significant atomic absorption by that element is absent. Zeeman Background correction may also be used.

7.9.7 Additives — Where additives are included in the solution preparations, including an ionization suppressant, details are provided in the procedures for the individual liquid chemical.

7.9.8 Remarks — The manufacturer's manual should be consulted for details on the venting of burner gases and for safety precautions in the operation of the instrument and burner. The user should check the performance of an instrument from time to time to assure that adequate sensitivity is being attained for the needs of the particular determination.

7.10 Determination of Trace Elements by Flame Emission Spectrometry — The purpose here is to describe in general terms how the technique should be used when specified in the procedures for an individual liquid chemical to assure that the specifications are met for one or more stated elements (commonly sodium or potassium, or both). No general procedure is given since operating details will vary with instrumental design; consequently, the manufacturer's manual should be followed.

7.10.1 Equipment — The flame photometer should have a monochromator with a resolution of at least 1 nm. The detector should have a red-sensitive phototube, a filter to remove the sodium D-line for potassium determinations, and a photomultiplier. (Instruments having a photomultiplier specially sensitive to the potassium line do not need a red-sensitive phototube.) The slit width must be adjustable. A sensitivity control should be available. Also, the burner should have controls for adjusting its position and gas flow rates. The read-out device may be a meter, digital display, or recorder.

7.10.2 Flame and Burner Conditions — The flame gases to be used are specified in the procedure for the individual liquid chemical. The burner position and gas flow rates should be optimized for a given determination.

7.10.3 Solution Conditions — The solutions are prepared as given in the procedures for the individual liquid chemical.

7.10.4 Standards — A stock standard solution for each relevant element should be accurately prepared, usually at a concentration of 0.1% wt/vol, as given either in the procedure for the individual liquid chemical or in §4. Working standard solutions should be prepared on the day of use by dilution of a stock standard solution.

7.10.5 Quantitation — Commonly, a single addition of standard is employed. It is applicable at low concentration where a linear relationship exists between signal and concentration. By this technique, the standard has a matrix similar to the sample.

7.10.5.1 The amount of standard to be added to the sample to make the standard solution is given in the procedures for the individual liquid chemical, and corresponds to the specification value of the element being determined.

7.10.5.2 For specific tests, the signals are measured for the sample solution and the standard solution. The instrument is optimized with the standard solution at the wavelength specified. It is then zeroed with water, and the sample is aspirated into the flame and its signal read. Without changing the slit, the wavelength is changed to a nearby setting and the background value determined. The difference between the background value and the value for the sample should not exceed the difference between the values obtained for the sample and standard solutions.

7.10.6 Remarks — The manufacturer's manual should be consulted for details on the venting of burner gases and for safety precautions in the operation of the instrument and burner. The user should check the performance of the instrument from time to time to assure that adequate sensitivity is being attained for the needs of the particular determination.

7.11 Determination of Trace Elements by Graphite Furnace Atomic Absorption Spectrometry — The purpose here is to describe in general terms how the technique should be used when specified in the procedures for an individual liquid chemical to assure that the specifications for one or more stated elements are met. No general procedure is given since operating details will vary with instrument design; consequently, the manufacturer's manual should be followed.

7.11.1 *Equipment* — The source is a hollow cathode lamp, or equivalent, for the relevant element. The furnace will contain a pyrolytic coated graphite tube that has been conditioned according to manufacturer's instructions. The monochromator should have a resolution of at least 0.1 nm. The signal from the photomultiplier tube detector is read from a meter, digital display, or recorder tracing. Zeeman or deuterium background correction can be used. An autosampler is suggested in order to obtain better reproducibility.

7.11.2 *Operating Conditions* — Use an acceptable flow rate (300 mL per minute) argon as the inert carrier gas, with no flow during the atomization portion of the program. The temperature programs outlined by the instrument manufacturers should be checked using known standards, and adapted as necessary for optimum performance for a specific instrument. Calibrate the temperature controller for the different atomization temperatures used for each element. The conditions for iron, potassium, calcium, and sodium are included in Table 9.

Table 9 Conditions for Graphite Furnace Atomic Absorption Spectrometry

<i>Element</i>	<i>Iron</i>	<i>Potassium</i>	<i>Calcium</i>	<i>Sodium</i>
Wavelength (nm)	248.3	766.5	422.7	589.1
Bandwidth (nm)	0.2	1.4	2.0	0.2
Current (mA)	30	6	8	8
L'vov Platform	Yes	Yes	Optional	Yes
Sample Size (μL)	20	10	10	10

7.11.3 *Solutions Conditions* — The solutions are prepared as given in the procedures for the individual liquid chemical. All solutions will be prepared in a clean environment as defined in the SEMI guidelines.

7.11.4 *Working Standard Solutions* — Working standard solutions should be prepared on the day of use by dilution of a stock standard solution.

7.11.5 *Sample Preparations* — Sample preparation will be analyzed neat, with pre-concentration, or by dilution, according to the individual procedure.

7.11.6 *Quantification* — Results shall be reported as the result of three replicate analyses. Each replicate is the result of an 8-second integration of the peak area with the instrument in concentration mode. Recording should commence 5 seconds before the atomization, and baseline should be established 2 seconds prior to atomization. Reading should commence 1 second prior to atomization. Alternate integration schemes for the analytical peak shall be performed in a manner that captures all of the signal, as appropriate for the instrument.

7.11.7 *Calibration* — Calibrate the instrument using a standard prepared in the appropriate grade of 18.2 Mohm-cm water that meets ASTM Standard Guide D5127, over a concentration range appropriate for the analysis. The blank must also be contained in the appropriate grade of 18.2 Mohm-cm water that meets ASTM Standard Guide D5127.

7.12 *Determination of Trace Elements by Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES)* — The purpose here is to describe in general terms how the technique should be used when specified in the procedures for an individual liquid chemical to assure that the specifications for one or more stated elements are met. No general procedure is given since operating details will vary with instrument design; consequently, the manufacturer's manual should be followed.

7.12.1 *Equipment* — A spectrometer with sufficient resolution to separate the typical spectral lines of the elements stated in the specification for an individual liquid chemical. The signal from the detector is processed by a computer system and displayed on a recorder with graphics capability. A background correction technique is required. Typical emission lines are listed in Table 10.

Table 10 Suggested Plasma Emission Line

<i>Element</i>	<i>Symbol</i>	<i>Line (nm)</i>
Aluminum	(Al)	396.15
Antimony	(Sb)	206.83
Arsenic	(As)	193.76
Barium	(Ba)	455.40

<i>Element</i>	<i>Symbol</i>	<i>Line (nm)</i>
Boron	(B)	249.77
Cadmium	(Cd)	214.44
Calcium	(Ca)	393.27
Chromium	(Cr)	205.55
Copper	(Cu)	324.75
Iron	(Fe)	238.20
Lead	(Pb)	220.35
Lithium	(Li)	670.78
Magnesium	(Mg)	279.55
Manganese	(Mn)	257.60
Nickel	(Ni)	231.6
Potassium	(K)	766.49
Sodium	(Na)	589.59
Tin	(Sn)	189.99
Titanium	(Ti)	334.94
Vanadium	(V)	292.40
Zinc	(Zn)	213.86

7.12.2 *Operating Conditions* — Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must be investigated and established for each individual analyte line on that particular instrument. Types of interferences other than the simple overlap of a concomitant line on an analyte line must be considered.

7.12.3 *Solution Conditions* — The solutions are prepared as given in the procedures for the individual liquid chemical.

7.12.4 *Working Standard Solutions* — Working standard solutions should be prepared on the day of use by dilution of a stock standard solution. For best results, standards should be matrix matched.

7.12.5 Multi-element standards may be prepared. The components should be selected to provide stable standards within the required concentration range.

7.12.6 *Quantitation* — Commonly, the samples are measured against matrix-matched calibration standards; however, whenever a new or unusual matrix is encountered, the method of standard addition is employed. The standard addition does not detect coincident spectral overlap; an alternate wavelength or an alternate method is recommended for verification. A calibration obtained with aqueous standards may be used if its slope and the standard addition method slope are identical.

7.12.7 *Evaporation of Sample* — The detection limits achievable can be improved by preconcentration of a large sample by evaporation. Through volatilization there may be a loss of trace elements, and recovery study data must be obtained. The loss of trace elements in this enrichment process can be reduced, often by the presence of additives. The evaporation must be conducted under “clean air” conditions, and reliability should be assured by use of a “blank” or control sample determination. The sample size will depend on the detection limits required and the capability of the instrument used.

7.12.8 *Background Correction* — A background correction technique is required for variable background contribution to the determination of trace elements. The correction interval should be selected such that the intensity of the background at that wavelength is equal to the background intensity under the analyte peak.

7.12.9 *Additives* — The procedures for the individual liquid chemical will provide detailed instructions for required additives in the sample solution.

7.12.10 *Remarks* — The manufacturer’s manual should be consulted for proper use of the instrument. The user should check the performance of the instrument from time to time to assure that adequate sensitivity is being attained for the needs of the particular determination.

7.13 Determination of Trace Elements by Inductively Coupled Plasma Mass Spectrometry (ICP-MS) — The determination of trace elements in liquid chemicals which are commonly used in advanced semi-conductor manufacturing requires a sensitive technique with the ability to perform simultaneous analysis for multiple elements. Achievable detection limits are adequate for sample analysis with little sample preparation prior to analysis. Liquid chemical matrix reduction needed to perform routine analysis is often achieved by dilution of the sample. Water used for dilution should be the appropriate grade of 18.2 Mohm-cm water that meets ASTM Standard Guide D5127. Matrix removal methods which require sample evaporation should be performed under HEPA filtered air such that reproducible blank samples are obtained at or less than one half the specified limit of trace elements for the sample. No specific procedure for inductively coupled plasma mass spectrometry is included since instrumentation and practices vary among laboratories. Some practical considerations relevant to the method are outlined below.

7.13.1 Equipment — A mass spectrometer with better than 1 amu resolution from 5–250 amu is required to separate the isotopes of the elements stated in the specification for an individual liquid chemical. The use of hot or cool plasmas as well as a collision/reaction cell may be required to meet the SEMI detection limit requirements for certain analytes. The signal from the detector is digitized by a computer system for quantitative calculation. Interference correction may be required for some isotopes. Typical analytically useful masses are listed in Table 11.

Table 11 Suggested Analytical Masses

<i>Element</i>		<i>Masses</i>
Aluminum	(Al)	27
Antimony	(Sb)	121, 123
Arsenic	(As)	75
Barium	(Ba)	137, 138
Boron	(B)	11
Cadmium	(Cd)	111, 114
Calcium	(Ca)	40
Chromium	(Cr)	52
Copper	(Cu)	63, 65
Iron	(Fe)	56
Lead	(Pb)	206, 207, 208
Lithium	(Li)	7
Magnesium	(Mg)	24
Manganese	(Mn)	55
Nickel	(Ni)	58, 60
Potassium	(K)	39
Sodium	(Na)	23
Tin	(Sn)	118, 120
Titanium	(Ti)	48
Vanadium	(V)	51
Zinc	(Zn)	64, 66

7.13.2 Sample Introduction — Conventional glassware used for sample introduction is in most cases appropriate for liquid chemical analysis, however for Grade 5 liquid chemicals an ultra clean sample introduction system is recommended. Liquid chemicals containing hydrofluoric acid (HF) require a HF resistant introduction system unless the hydrofluoric acid matrix is removed prior to the analysis.

7.13.3 Ionization Source — An inductively coupled plasma using argon as the support gas has proven to be satisfactory as an ionization source for the method. Ionization conditions present in the argon plasma are highly dependent on the various argon gas flow rates used to support the plasma. Sample introduction techniques used to introduce the sample into the plasma are important to the analysis of the samples and must be optimized to allow successful analysis. In particular, sample uptake and nebulizer gas flow require special attention and should be treated carefully in order to optimize instrument performance for each of the various liquid chemicals.

7.13.4 Evaporation of Samples — Liquid chemical matrix elements may be the source of spectral interferences on certain target elements e.g. 48SO interferes with 48Ti in a sulfuric acid matrix when using quadrupole-based ICP-MS. Such spectral interferences may be significantly reduced or eliminated by controlled evaporation of the sample followed by dissolution and analysis in ultrapure nitric acid or other suitable solvent.

7.13.5 Solution Conditions — The solutions are prepared as given in the procedures for the individual liquid chemicals or elements.

7.13.6 Working Standard Solutions — Working standard solutions are prepared daily by dilution of stock standard solutions. Internal standard solutions, if used, are prepared similarly.

7.13.7 Quantification — Quantification may be performed by the method of standard additions or by external calibration. External standards may also be matrix matched in ultra pure liquid chemicals to assure that plasma conditions during the analysis are similar for both standards and samples. For those liquid chemicals, which require matrix removal, the preferred matrix after preparation is dilute nitric acid (typically 1–3%.) The working standard solutions are prepared to cover the expected range of trace element concentration. Background subtraction by use of a reagent blank solution is may be used for sample analysis by this technique.

7.13.8 One or more internal standards (such as indium or rhodium) may be used for drift correction during the analysis.

7.13.9 Remarks — The manufacturer's manual should be consulted for details specific to the operation of the instrument. Performance checks on the instrument should be made from time to time to assure adequate mass resolution, performance, and sensitivity.

7.14 Calibration and Measurement Method for Particles in Liquids — This standard describes the apparatus and methods used to calibrate optical particle counters (OPCs) and to count hard particles in liquid reagents. The procedures in this standard provide a means of comparison of particle levels in various reagents using pressurized sampling and counted using the various available instruments. While the procedures in this standard are primarily directed at off-line sampling or sampling from containers, OPCs can also be used for continuous on-line particle measurement. While some substantial technical difficulty may accompany the implementation of the pressurized sampling technique for containers of more than 10 L, the method should be followed as closely as possible to maximize the integrity of the data. The following procedures are partially based on the publications in the attached bibliography, which should be consulted for further details.

7.14.1 Apparatus — The apparatus used to measure the concentration of particles in reagents is comprised of a sample supply system, a particle counter, and a flow measurement and control system. Two manual flow measurement and control systems are presented in Figures 4 and 5. Several manufacturers have developed automated pressurized sampling systems.

7.14.2 Discrete Sample Supply System — In the method specified in this standard, the pressure in the reagent container is raised sufficiently above atmospheric pressure to produce the required flow and to reduce microbubble formation, which causes falsely high counts. Pressurizing containers of reagents presents a substantial safety hazard. Several systems have been developed for pressurizing the contents of a reagent container without placing a pressure differential across the reagent container wall. A pressurized sampling system is required for all reagents.

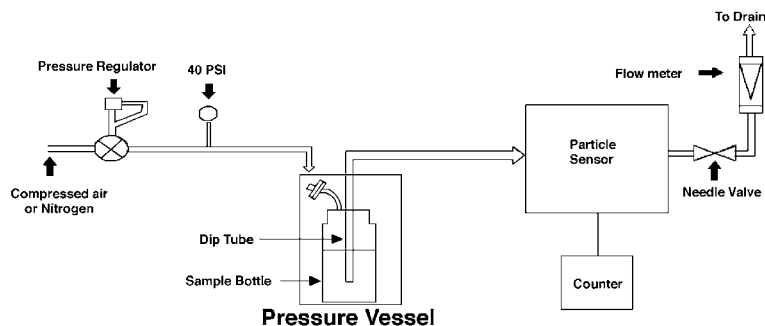


Figure 5
Pressurized Test System

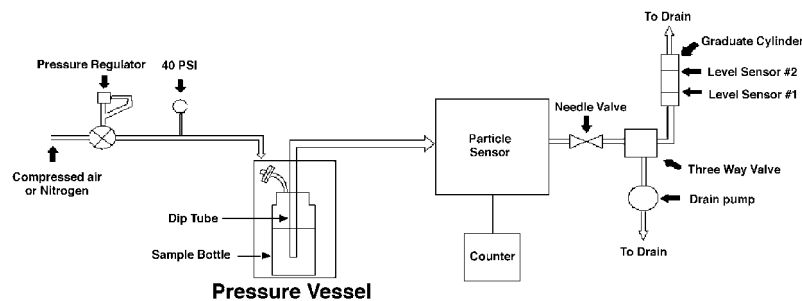


Figure 6
Pressurized Test System

7.14.2.1 The reagent container is placed in a canister suitable for use as a pressure vessel at 4 bar (60 psi), such as a large steel filter housing. The interior of the canister should be resistant to corrosion by the reagents being tested. This canister is fitted with a relief valve set at 3.5 bar (50 psi), a nitrogen inlet, and a sealed feed-through for the sample tube. The reagent container is fitted with a perfluoroalkoxy (PFA) cap to which is attached a hydrophobic polytetrafluoroethylene (PTFE) nitrogen inlet filter. This permits the nitrogen pressures inside and outside the reagent container to equilibrate when the vessel is pressurized and prevents particles from the pressure canister from entering the sample reagent container.

7.14.2.2 Either of two sample and dip tube configurations may be used. In the first, a PFA dip tube, which extends to 5 cm from the bottom of the reagent container, is attached to the cap. The sample tube, which consists of a single continuous piece of PFA tubing to reduce the chance of incidental contamination during measurement, is connected to the cap and the particle counter. This configuration has the advantage that it allows the sample tube to be disconnected from the cap and dip tube, reducing the risk of splashing and of cross-contaminating samples. The disadvantage of placing a fitting between the sample and the counter is that the fitting may compromise the integrity of the sample.

7.14.2.3 In the second configuration, a single tube, without valves, fittings, or discontinuities extends from the counter, through the cap to 5 cm from the bottom of the reagent container. This has the advantage that it ensures the integrity of the sample but the disadvantage is that the risk of splashing and cross-contamination is increased.

7.14.3 *Particle Counter* — Laser light scattering OPCs from any of several suppliers may be used provided they are in calibration, and used subject to the limitations described in ¶7.14.3.1.

7.14.3.1 *Limitations on Counting Particles in Liquid Reagents using OPCs* — OPCs are calibrated using polystyrene latex (PSL) beads suspended in high purity water. The optical properties of naturally occurring particles in liquid reagents are different from the optical properties of PSL and water. OPCs detect “light-scattering centers” and calculate particle size and concentration based on the assumption that the light-scattering centers are equivalent to PSL in water. In reality, light scattered by a particle is dependent upon particle size, particle shape and the difference between the particle’s refractive index and the refractive index of the surrounding medium. When properly calibrated OPCs from different manufacturers are used to measure particles in high purity water, they give comparable size and concentration data.

7.14.3.1.1 Commercially, OPCs are manufactured with different illuminating wavelengths and a variety of optical arrangements for collecting light scattered by particles. Because of the significant difference in the refractive index of particles relative to the refractive index of water, the resulting scattered light profile is readily detected by all OPCs. Hence there is relatively good comparative agreement between OPCs when used to detect particles in high purity water.

7.14.3.1.2 When calibrated OPCs are used to measure particles in semiconductor liquid reagents, a totally different situation is encountered. The refractive index of liquid reagents varies significantly from water and is, in general, greater than water. While the refractive index of reagents is known and can be adjusted for, the refractive index of particles is unknown and, in many circumstances, is expected to be very close to the refractive index of reagents.

Particles therefore have a tendency to become invisible to an OPC. The degree of invisibility will depend on the optical arrangement of each OPC. A study carried out in 1999⁸ found that particle concentrations reported by different sensors varied significantly. Chemical-to-chemical comparison indicated that there was no sensor that consistently reported higher or lower concentrations than other sensors.

7.14.3.2 Using OPCs in Semiconductor Liquid Reagents — OPCs have a critical role to play in monitoring particles in liquid reagents. OPCs can detect low concentrations of small particles in real-time and can be easily calibrated using PSL beads. However, the current state of OPC technology limits their use to claim that a liquid reagent meets a standard particle concentration at a particular particle size.

7.14.3.2.1 When a supplier of a liquid reagent uses an OPC to specify that reagent's particle concentration, for example on a Certificate of Analysis, the make and model of OPC must also be specified. If the customer of the liquid reagents wishes to check whether the incoming reagent meets the claimed level of cleanliness, the customer must use an OPC of the same make and model number. However, it has also been shown that even two calibrated "identical" OPCs can differ by 10–20% when measuring particles in liquid reagents.

7.14.4 Flow Control and Measurement System — A discrete or a continuous method may be used to control and measure the flow of the sample through the particle counter. In both procedures, a needle valve, downstream of the counter, is used to control the flow rate. In the discrete procedure, a measured volume of fluid is counted and collected in a graduated container. Automatic level detection may be used to start and stop the counter. In the continuous procedure, the flow rate is set and the count is accumulated for a measured time period.

7.14.5 Calibration — Each particle counter (sensor and signal analysis unit) must be calibrated at least once every twelve months.

7.14.5.1 It is recommended that the calibration be performed by the instrument manufacturer or a metrology laboratory certified by the instrument manufacturer. Calibration is performed by counting polystyrene latex (PSL) beads suspended in water. PSL beads of known size (characterized by transmission electron microscopy (TEM) and concentration (characterized by a scanning electron microscope SEM) must be used. In addition most instrument manufacturers use a working standard or "gold standard" OPC as a reference against which all OPCs of the same Model are compared.

7.14.5.2 The determination of size correction factors for particle counters is not recommended. Particle counters should be calibrated with PSL beads in accordance with the manufacturer's recommendation.

7.14.6 Coincidence Correction Factors — The determination of coincidence correction factors for particle counters is not recommended. Particle counters should be calibrated with PSL beads as per the manufacturer's recommendation. Coincidence correction factors are only valid for monodispersed concentration of particles.

7.14.7 Efficiency Correction Factors — The determination of efficiency correction factors for particle counters is not recommended. Particle counters should be calibrated with PSL beads as per the manufacturer's recommendation.

7.14.8 Monthly Check — It is desirable to check the OPCs size calibration once a month. PSL beads of known size and approximate concentration can be purchased from several suppliers. Prepare a stock suspension of a combination of about 10^7 particles/L of each of the three sizes of PSL beads. Several solutions have been found to provide acceptably stable suspensions. Among them are: 25:1 ethanol/: water, and water with 0.01% surfactant and 0.05% bactericide. Dilute 10 mL of stock suspension to 1 L and measure the particle concentration using the procedure specified for reagents. If the results of this check are not within 10% of the expected PSL bead size, the OPC shall be recalibrated.

7.14.9 Measurement Procedure

7.14.9.1 Setup — Gently invert the sample reagent container three times to homogenize the particle distribution, then set it aside for 30 minutes to remove the bubbles formed during mixing. Under no circumstances must the sample reagent container be vigorously shaken or sonicated as this would generate numerous particles.

⁸ Carrieri, D., D.C. Grant and W. Kelly, "A Comparison of Optical Particle Sensors Used to Measure Particle Concentration in High-Purity Process Chemicals" Proceedings SEMICON West 1999

7.14.9.2 The smallest particle counter threshold should deliver at least 50% particle counting efficiency at the control size limit. The control size limit is the particle size agreed upon between supplier and customer that represents an acceptable size level. In general this size will be between 0.1 and 0.5 μm .

7.14.9.3 Attach the cap with the gas inlet filter and sample tube to the reagent container to be sampled. Place the reagent container in the canister, and seal the canister so that it may be pressurized. With the sample flow control valve open, gradually (< 3 bar/min) pressurize the sample to 3 bar for H_2O_2 and NH_4OH or as necessary to obtain the required flow for other fluids. Allow at least 250 mL of the sample to pass through the sensor before counting.

7.14.9.4 For each sample, after 5 aliquots have been measured, perform a chi-squared test to determine if the data are statistically valid. If the chi-squared test fails, examine the apparatus for defects, such as loose or cracked fittings, repair as necessary, and repeat the counting of 5 aliquots. If the sample fails both the second and third chi-squared tests, it is considered to be immeasurable and should be discarded. If a second sample gives similar results, the apparatus, including the OPC, may be defective and should be checked with a well-characterized sample and repaired if necessary.

7.14.10 *Measurement*

7.14.10.1 *Discrete Sampling* — Measure multiple aliquots of the same sample. The flow rate must be the same as that at which the instrument was calibrated. The statistical significance of any count data must be taken into consideration. Some OPCs inspect as little as $1/1000^{\text{th}}$ of the available sample flow, and may require a greater number of aliquots and larger sampling volume to ensure good statistics. For example, to obtain a coefficient of variation (standard deviation/mean) of 10%, it is necessary that at least 100 particles greater than the control size limit be counted.

7.14.10.2 *Continuous Sampling* — If continuous sampling apparatus is being used, adjust the needle valve on the rotameter to obtain a flow rate consistent with the instrument manufacturer's recommendations. This flow rate must be the same as that at which the instrument was calibrated. Correct the indicated value for the effect of the fluid density and viscosity on the rotameter reading. Allow the first minutes' fluid to flow to drain, then count the particles in each of five, 15 second intervals. The statistical significance of any count data must be taken into consideration. Some OPCs inspect as little as $1/1000^{\text{th}}$ of the available sample flow, and may require a greater number of aliquots and larger sampling volume to ensure good statistics. For example, to obtain a coefficient of variation (standard deviation/mean) of 10%, it is necessary that at least 100 particles greater than the control size limit be counted.

7.14.11 *Shutdown* — As in all portions of this procedure, appropriate safe liquid chemical handling practices must be used. In particular, attention should be paid to the reactions between different reagents and between reagents and diluents, including water, and to the inherent hazards of these materials.

7.14.11.1 After the measurement has been completed, vent the pressure vessel to atmospheric pressure through an appropriate scrubber or exhaust hood, then flush the vessel with nitrogen to remove residual reagent vapors.

7.14.11.2 Flush the OPC and sample system with water or an appropriate solvent to remove the remaining fluid.

7.14.12 *Coincidence and Efficiency Corrections* — The determination of coincidence and efficiency correction factors for particle counters is not recommended. If an OPC's maximum concentration is exceeded, a different OPC should be selected, or the sample diluted. However any sample dilution will impose additional error and is not recommended.

7.14.13 *Index of Refraction Correction* — The determination of an index of refraction correction factor for particle counters is not recommended, as the refractive index of the contaminating particle is unknown.