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DRAFT

Tier 2 Critical Procedures Manual

Technikon # 1411-624

August 2005
(Revised for public distribution, September 2005)

United States Environmental Protection Agency

CISA

AMERICAN FOUNDRY SOCIETY, INC.

UNITED STATES COUNCIL FOR AUTOMOTIVE RESEARCH
USCAR
DAIMLERCHRYSLER Ford Motor Company General Motors

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TECHNIKON LLC

CRITICAL

PROCEDURES

MANUAL

Technikon # 1411-624

Copy No. 1

TECHNIKON LLC

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CRITICAL PROCEDURES MANUAL				CPM-1001
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	C. R. Glowacki	1	1	NEW

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MANUAL CONTROL				
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	C. R. Glowacki	1	4	NEW

MANUAL CONTROL

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MANUAL TITLE				Document #
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DISTRIBUTION

Master Copy	No. 1	Technikon ISO Library	ISO-9001 Coordinator or designate
	No. 2		
	No. 3		
	No. 4		
	No. 5		
	No. 6		
	No. 7		
	No. 8		
	No. 9		

This manual outlines Technikon LLC critical procedures and is intended to guide Technikon's continuous improvement process.

_____

C. R. Glowacki
ISO-9001 Coordinator

First Published –

MANUAL REVIEW AND REVISION

The ISO Coordinator or his designate has the authority to issue and approve revisions of this manual. The manual will be reviewed at least annually in a manner selected by the ISO Coordinator. The requirements outlined in Section 5 of the Quality Manual govern the format and change/modification procedure for this manual.

Changes made to individual pages do not require reissue of the entire section. Each page will note the latest revision date. Changes will be identified by an (*) in the left hand margin. Hand written changes or updates are not permitted except in emergencies and only with the authorization of the ISO Coordinator. Dates and initials are required adjacent to those changes. Any suggestions for changes or modifications should be sent to the ISO coordinator.

The ISO Coordinator sends out proposed changes for review to the ISO Implementation Committee. The Committee will assess the effect, if any, these changes may have on their quality systems. Any change to this manual that affects the Quality System will be communicated to the ISO Coordinator for resolution.

The ISO Coordinator directs the disposition of obsolete copies of the manual, sections, or pages being revised. Copying of the manual is permitted; all such copies are readily identifiable as non-controlled copies. Controlled copies of this manual are printed on paper with a colored "Technikon" logo. The distribution of controlled copies is shown in Document CPM-1002, of this Manual.

REVISION AND REVIEW HISTORY

See Critical Procedures Document CPM-1005, Section 4.4.4.

TECHNIKON LLC

MANUAL TITLE				Document #
CRITICAL PROCEDURES MANUAL				CPM-1003
DOCUMENT TITLE				ISSUE DATE
INTRODUCTION				
APPROVED BY	PREPARED BY:	PAGE	OF	SUPERCEDES
	C. R. Glowacki	1	1	NEW

This manual defines Technikon's critical procedures with regard to the application of quality systems in all areas and activities having an influence on quality of services and customer satisfaction.

This manual is set out as a reference document outlining both the critical procedures of Technikon and references to Technikon's controlled documents that comply with the ISO 9001 - 2000 standard.

Individual document reference numbers are quoted within the body of this manual and references will be made to locations where these documents or records may be found. (Also, refer to the Index located in Appendix I of the Quality Manual). This permits the revision of procedures in various work departments of Technikon independent of the Critical Procedures Manual.

The Critical Procedures Manual and procedures of various work departments may contain specific procedures and references that are proprietary and, therefore, considered **Confidential**. Access to these documents will be granted only when authorized by the Technikon President or designate(s).

The "Scope of Registration" encompasses provision of a service of emission testing and process improvement including contract design of processes for casting related industries.

The functions of the manual are to

1. Define Technikon's critical procedures regarding quality in a form directly related to ISO-9001 and equivalent standards.

2. Identify and reference the critical procedures and records that affect quality. (Refer to Index of Department Manuals located in Appendix I of the Quality Manual for specific procedures by Technikon department.)

3. Act as the reference document for purposes of audit, management review and external assessment of Technikon's quality program.

TECHNIKON LLC

MANUAL TITLE				Document #
CRITICAL PROCEDURES MANUAL				CPM-1004
DOCUMENT TITLE				ISSUE DATE
DEFINITIONS				
APPROVED BY	PREPARED BY:	PAGE	OF	SUPERCEDES
	C. R. Glowacki	1	1	NEW

CPM	Critical Procedures Manual
AD	Administration Department
OP	Operations Department
MT	Measurement Technologies Department
EHS	Environmental Health and Safety Department

TECHNIKON LLC

MANUAL TITLE				Document #
CRITICAL PROCEDURES MANUAL				CPM-1005
DOCUMENT TITLE				ISSUE DATE
DOCUMENT CONTROL				
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	C. R. Glowacki	1	9	NEW

ISO-9001 REFERENCE

1. PURPOSE

To define the methods used to control the issuance, correction, revision, and distribution of quality documents that are essential to the integrity of the Technikon Quality System.

2. SCOPE

Document control shall be addressed by the following applicable systems:

2.1 Issuance and approval of original documents.
2.2 Distribution of documents.
2.3 Identification of controlled and uncontrolled/obsolete documents.
2.4 Correction and/or revision of documents.
2.5 Document status.
2.6 Electronic documents.

3. DEFINITIONS

AD = Administration
MT = Measurement Technology
OP. = Operations
EHS = Environmental Health and Safety Department
ISO Coordinator = ISO Coordinator or designate
Technikon Quality System Manuals:
(1st tier) Quality Manual
(2nd tier) Critical Procedures Manual
(3rd tier) Department Policy & Procedures Manuals
(4th tier) Forms Manual
Internal Audit Binder
Management Review Binder
Vendor Deficiency Binder

ISO-9001 REFERENCE

4. PROCEDURE - DOCUMENT CONTROL

4.1. Approval and Issuance of Original Documents.

4.1.1. **Quality Manual.** The ISO Coordinator has responsibility for the approval and issuance of Quality Documents contained in the Quality Manual.

4.1.1a These documents shall be reviewed for accuracy and applicability to the Technikon Quality System by the ISO Coordinator. Approval for issuance shall be by signature of the ISO Coordinator on a cover page containing the document issue and revision number on the line labeled “Approved by:”. Upon approval, a copy of the document will be issued to each party specified on the distribution list.

4.1.2. **Critical Procedures Manual.** The ISO Coordinator has responsibility for the approval and issuance of Quality Documents contained in the Critical Procedures Manual.

4.1.2a These documents shall be reviewed for accuracy and applicability to the Technikon Quality System by the ISO Coordinator. Approval for issuance shall be by signature of the ISO Coordinator on a cover page containing the document issue and revision number on the line labeled “Approved by:”. Upon approval, a copy of the document will be issued to each party specified on the distribution list.

4.1.2b These documents shall be issued with a unique number assigned by the ISO Coordinator. This number shall start with the prefix CPM and be allowed the numeric range of 1000 to 1999.

ISO-9001 REFERENCE

4.1.3. **Department Policy and Procedures Manual.** The ISO Coordinator has responsibility for the final approval and issuance of Quality Documents contained in each Department Policy and Procedures Manual.

4.1.3a These documents shall be reviewed for accuracy by the responsible Department Head. Initial approval is necessary before submittal to the ISO Coordinator. Initial approval shall be by signature of the specific Department Head on the Manual Control page near the beginning of the manual. Approval of revisions shall be by signature of the respective Department Head in the appropriate box on Technikon Form #007 accompanying the revision.

4.1.3b After approval by the respective Department Head, these documents shall be reviewed for accuracy and applicability to the Technikon Quality System by the ISO Coordinator. Final approval for issuance shall be by signature of the ISO Coordinator in the box labeled “Approved by :”of the cover page header. Upon approval, a copy of the document will be issued to each party specified on the distribution list.

4.1.3c These documents shall be issued with a unique number assigned by the ISO Coordinator and according to the following parameters:

ISO-9001 REFERENCE

- **Administration Department Policy and Procedures Manual** - Each document number shall start with the prefix "AD" to be followed by a unique numeric designation in the range of 2000 to 3999.

- **Operations Department Policy and Procedures Manual** - Each document number shall start with the prefix "OP" to be followed by a unique numeric designation in the range of 4000 to 5999.

- **Measurement Technologies Department Policy and Procedures Manual** - Each document number shall start with the prefix "MT" to be followed by a unique numeric designation in the range of 6000 to 7999.

- **Environmental Health and Safety Department Policy and Procedures Manual** - Each document number shall start with the prefix "EHS" to be followed by a unique numeric designation in the range of 8000 to 9999.

4.1.4. **Forms Manual**. The ISO Coordinator has responsibility for the final approval and issuance of Quality Documents contained in the Forms Manual. Only those forms unique to the Technikon Quality System and under its direct control shall be included in the Forms Manual

ISO-9001 REFERENCE

4.1.4a These documents shall be reviewed for accuracy and applicability to the Technikon quality system and will be added to the Forms Manual and the template in *(Current network path to be added)* will be updated

4.1.5. Any member of the Technikon Quality System may initiate a quality document.

4.2. Distribution of Documents.

4.2.1. The ISO Coordinator shall be responsible for the distribution of controlled Quality System documents.

4.2.2. Master Distribution List. A distribution list of all controlled quality documents shall be maintained in a separate binder labeled “Master List” in a section entitled “Controlled Document Distribution List” and kept in the Technikon ISO library

4.2.2a The Master Distribution List will be maintained by the ISO Coordinator and be comprised of each individual Quality System Manual Distribution List located in separate sections.

4.2.2b The Quality System “Master Distribution List” shall be reviewed at least annually for completeness and accuracy.

4.2.2c Upon review and approval of the Master Distribution List and Controlled Document Distribution List, a copy of each Quality Manual Distribution List shall be signed by the ISO coordinator and issued, with the revision number and date, to the appropriate responsible party specified on the Master Distribution List.

ISO-9001 REFERENCE

4.2.3. Each Quality System Manual shall contain a section entitled "Distribution" to be found in the "Manual Control" document and will be listed in a table of contents

4.2.3a This section shall contain the following:

- Copy Numbers
- Copy Holders
- Approval signature of ISO Coordinator
- Issue and Supersede Date

4.3. Identification of Controlled and Uncontrolled/Obsolete Documents.

4.3.1. All documents, paper and electronic, that are contained in Technikon Quality System Manuals shall be controlled. A master list of all controlled documents shall be found in a section entitled "Controlled Document List" in the "Master List" binder located in the Technikon ISO Library and will be maintained by the ISO Coordinator.

4.3.1a Controlled paper documents shall be identified by the colored "Technikon" logo in the top left-hand corner of the document in compliance with Form #XXX of the Forms Manual. Any variation or incompleteness of this description will invalidate the document's controlled status and subsequently shall be subject to the conditions set forth in section 4.3.3 of this document.

4.3.1b Electronic controlled documents shall be located solely in the *(Add current network path)* folder and be made accessible only to the ISO Coordinator or designate by a security password.

4.3.2. All Technikon Quality System Manuals shall contain original documents. No photocopies or facsimiles of any kind will be permitted.

ISO-9001 REFERENCE

4.3.3. Uncontrolled copies of controlled documents shall not be used within the Technikon Quality System except for training, distribution to customers, or draft revisions.

4.3.4. A document shall be considered "obsolete" when replaced by a new or revised version to the Technikon Quality System. The document will be taken out of the Technikon Quality System as outlined in section 4.4.1c of this document.

4.4. <u>Document Review, Correction, and/or Revision</u>

4.4.1. Document correction or revision may be initiated by any member of the Technikon Quality System.

4.4.1a Approval of corrected or revised documents will follow the guidelines set forth in Section 4.1 of this document

4.4.1b Only uncontrolled copies may be used as draft materials for document correction or revision.

4.4.1c Document revision distribution will include a copy of Technikon Form #XXX to be completed and returned to the ISO Coordinator by the responsible party upon insertion of the new or revised document into the Technikon Quality System Manual and destruction of the obsolete document. The responsible party must comply within 30 days.

4.4.2. All 1st & 2nd tier Technikon Quality System Documents shall be reviewed at least annually by the ISO Coordinator for completeness and accuracy.

4.4.2a Approval of corrected or revised documents will follow the guidelines set forth in Section 4.1 of this document.

ISO-9001 REFERENCE

4.4.3. Each 3rd tier Department Policy and Procedures Manual document shall be reviewed at least annually by the respective Department Head for completeness and accuracy.

4.4.3a Approval of corrected or revised documents will follow the guidelines set forth in Section 4.1.3 of this document

4.4.4. All revisions to Technikon Quality System Manuals will be followed up by an entry in the "Review and Revision" section of the respective Quality Manual Master Copy as stated in Section 4.5.2 of this document.

4.5. Document Status

4.5.1. The Master Copy of each Quality System Manual shall contain a section entitled "Review and Revision", which will contain a review history using Technikon Form #XXX of the Forms Manual. The "Review and Revision" section will be designed in such a way that each Quality System document will have its own unique tabbed area that is categorized in a table of contents at the beginning of the section.

4.5.1a Technikon Form #XXX of the Forms Manual shall be sectioned to contain lists of document revisions where each entry will include:

- Revision Number
- Revision Date
- Space for revision explanation and results
- Initial box for each responsible party on distribution list.
- Initial box entitled "Electronic Backup

ISO-9001 REFERENCE

4.5.2. The replacement of a controlled (paper) document, outlined in section 4.4.1c of this document, shall be verified by the ISO Coordinator's entry on the line corresponding to the responsible party in the appropriate Quality System Manual Master Copy "Review and Revision" entry for that document.

4.5.3. The replacement of a controlled (electronic) document, which constitutes revision of that document located in the *(Enter current network path)* folder and its backup, shall be verified by the ISO Coordinator's entry on the line marked "Electronic Backup" in the appropriate Quality System Manual Master Copy "Review and Revision" entry for that document.

4.6. Electronic Documents

4.6.1. The ISO Coordinator shall be responsible for electronic backup of controlled documents.

4.6.1a Electronic controlled documents shall be located solely in the *(Enter current network path)* folder and be made accessible only to the ISO Coordinator, Asst. ISO Coordinator, and designate(s).

4.6.2. Electronic controlled documents shall be backed up on CD or other media and stored in a binder named "Technikon Quality System Electronic Backup" and kept in the Technikon ISO library.

4.6.2a Electronic backup shall be performed after each document revision is issued.

TECHNIKON LLC

MANUAL TITLE				Document #
CRITICAL PROCEDURES MANUAL				CPM-1006
DOCUMENT TITLE				ISSUE DATE
CUSTOMER INTERFACE				
APPROVED BY	PREPARED BY:	PAGE	OF	SUPERCEDES
	C. R. Glowacki	1	4	NEW

ISO-9001 REFERENCE

1. PURPOSE

To define the manner in which Technikon receives requests for routine testing and process evaluation from its customers, tracks the progress of a test, and distributes the report to the customer.

2. SCOPE

Applicable to the following Customer Interface.

2.1 Emission Testing
2.2 Process Evaluation
2.3 Commercial Activities

3. DEFINITIONS:

None

4. PROCEDURE

4.1. Emission Testing – Scope 2.1

4.1.1. When a product is identified for routine testing under the CERP program, a Test Approval Form (Form #XXX) is completed by a member of the Technikon Quality System.

4.1.2. The draft Test Approval Form is reviewed by the Vice President – Operations and the Vice President – Measurement Technologies and signed to document approval.

4.1.3. The signed form is sent to the Manager – Process Engineering who drafts a Technikon Test Plan (Form #XXX).

4.1.4. The draft Test Plan is reviewed by the Vice President – Operations and the Vice President – Measurement Technologies and signed to document approval and returned to the Manager – Process Engineering with a copy going to the Manager – Laboratory Services.

ISO-9001 REFERENCE

4.1.5. The Manager – Process Engineering or designate prepares Process Instructions for the test. The Process Instructions are reviewed by the Vice President – Operations.

4.1.6. The Manager – Laboratory Services or designate prepares a Test Sampling Plan for the test. The Test Sampling Plan is reviewed by the Vice President – Measurement Technologies.

4.1.7. Following completion of the test a report is prepared. See CPM-1013.

4.2. Process Development – Scope 2.2

4.2.1. When a process is identified for routine evaluation or testing under the CERP program a Test Approval Form (Form #XXX) is completed by a member of the Technikon Quality System.

4.2.2. The draft Test Approval Form is reviewed by the Vice President – Operations and the Vice President – Measurement Technologies, if the process evaluation includes emission testing, and signed to document approval.

4.2.3. The signed form is sent to the Manager – Process Engineering who drafts a Technikon Test Plan (Form #XXX).

4.2.4. The draft Test Plan is reviewed by the Vice President – Operations and the Vice President – Measurement Technologies, if the Test Plan includes emission testing, and signed to document approval and returned to the Manager – Process Engineering with a copy going to the Manager – Laboratory Services, if emission testing is included in the plan.

4.2.5. The Manager – Process Engineering or designate prepares Process Instructions for the test. The Process Instructions are reviewed by the Vice President – Operations.

ISO-9001 REFERENCE

4.2.6. The Manager – Laboratory Services or designate prepares a Test Sampling Plan for the test, if emission testing is included in the test plan. The Test Sampling Plan is reviewed by the Vice President – Measurement Technologies.

4.2.7. Following completion of the test a report is prepared. See CPM-1013.

4.3. Commercial Activities – Scope 2.3

4.3.1. When a client contacts Technikon to conduct a routine commercial test or other routine project, the Vice President – Measurement Technologies or the Vice President – Operations gathers the pertinent information needed to define the test or project and prepares a proposal including estimated costing.

4.3.2. The draft proposal is submitted to the client for comment and/or approval.

4.3.3. Comments are included in a revised proposal that is submitted to the client for approval.

4.3.4. Work on the proposed test or project does not begin before a purchase order is received from the client.

4.3.5. The draft Technikon Test or Project Plan (Form #XXX) is prepared by the Manager – Process Management.

4.3.6. The draft Plan is reviewed by the Vice President – Operations and the Vice President – Measurement Technologies, if the Test Plan includes emission testing, and signed to document approval and returned to the Manager – Process Engineering with a copy going to the Manager – Laboratory Services, if emission testing is included in the plan.

4.3.7. The Manager – Process Engineering or designate prepares Process Instructions for the test or project. The Process Instructions are reviewed by the Vice President – Operations.

ISO-9001 REFERENCE

4.3.8. The Manager – Laboratory Services or designate prepares a Test Sampling Plan for the test, if emission testing is included in the test plan. The Test Sampling Plan is reviewed by the Vice President – Measurement Technologies.

4.3.9. Following completion of the test a report is prepared. See CPM-1013.

TECHNIKON LLC

MANUAL TITLE				Document #
CRITICAL PROCEDURES MANUAL				CPM-1007
DOCUMENT TITLE				ISSUE DATE
PERSONNEL TRAINING				
APPROVED BY	PREPARED BY:	PAGE	OF	SUPERCEDES
	C. R. Glowacki	1	3	NEW

ISO-9001 REFERENCE

1. PURPOSE

To ensure that members of the Technikon Quality System are provided with the necessary training to perform all activities affecting quality.

2. SCOPE

2.1. **Technikon Quality System** training shall be addressed through the following applicable systems:

2.1.1. Training to the ISO 9001 Standards
2.1.2. Tier I & II training
2.1.3. Tier III training

3. DEFINITIONS

Tier I = Quality Manual policies
Tier II = Critical Procedures Manual policies
Tier III = Each Department's Policy and Procedures Manual
ISO Coordinator = ISO Coordinator or designate

4. PROCEDURE - Training to ISO 9001 Standards

4.1. **The ISO Coordinator** has responsibility for training all Technikon members to the ISO 9001 Standards.

4.2. **All Members** of the Technikon Quality System will be required to complete training to the ISO 9001 Standards. Training shall consist of:

4.2.1. Understanding each ISO 9001 standard as it is stated by the ISO Coordinator.

4.2.2. Completing Technikon Form #XXX upon understanding the ISO 9001 Standards.

4.2.3. Return Technikon Form #XXX to the ISO Coordinator.

ISO-9001 REFERENCE

4.3. All ISO 9001 training records shall be maintained by the ISO Coordinator. These records shall consist of the completed Technikon Form #XXX for each member of the Technikon Quality System and shall be located in a binder labeled "Technikon Training Records".

5. PROCEDURE - Tier I & II Training

5.1. **The ISO Coordinator** has responsibility for Tier I & Tier II training of all Technikon employees.

5.2. **All members** of the Technikon Quality System will be required to complete Tier I & II training. Training shall consist of:

5.2.1. Reading and understanding each policy as it is stated in The Quality Manual and The Critical Procedures Manual.

5.2.2. Completing Technikon Form #XXX upon reading and understanding The Quality Manual and The Critical Procedures Manual.

5.2.3. Return Technikon Form #XXX to the ISO Coordinator.

5.3. **All Tier I & Tier II** training records shall be maintained by the ISO Coordinator. These records shall consist of the completed Technikon Form #XXX for each member of the Technikon Quality System and shall be located in a binder labeled "Technikon Training Records".

6. PROCEDURE - Tier III Training

6.1. **Each Department Head** has responsibility for the appropriate Tier III training of each member of their respective departments.

6.2. **Each Department Policy and Procedures Manual** shall contain a policy to address Tier III training as it applies to that department and shall consist of at least the following:

ISO-9001 REFERENCE

6.2.1. Procedure for the appropriate Tier III training of department members.

6.2.2. A method to verify and record that proficiency training was accomplished by each member in their respective areas where the activity of that member may affect quality.

6.2.3. Location of training records must be in a similar location as the respective Tier III Manual and that location must be identified in the procedure.

7. NEWLY HIRED PERSONNEL

7.1. **All Temporary Employees Having 720 Hours** or more of accumulated time in Technikon and all newly hired regular employees shall comply with Sections 4.2, 5.2, and 6.2 of this document before performing any quality related activities.

7.2. **Temporary Employees With Less Than 720 Hours** of accumulated time in Technikon shall be under the direct supervision of qualified personnel and shall not have any quality related activities. They shall receive, as a minimum, Tier III training as appropriate to their duties as determined by the respective Department Head.

8. PROCEDURE FOR JOB GROWTH

8.1. **Continuous Improvement** and job growth is addressed through the yearly appraisal process.

8.2. **Appraisals are Conducted** by a department head with an employee to set goals for additional training and job improvements. The goals are documented in the appraisal form.

8.3. **Appraisal Forms for Employees** are retained in the Technikon Administration Department.

TECHNIKON LLC

MANUAL TITLE			Document #
CRITICAL PROCEDURES MANUAL			CPM-1008
DOCUMENT TITLE			ISSUE DATE
INTERNAL AUDITING			
APPROVED BY	PREPARED BY:	PAGE OF	SUPERCEDES
	C. R. Glowacki	1 7	NEW

ISO-9001 REFERENCE

1. PURPOSE

To provide an internal quality auditing system that verifies Technikon compliance with ISO-9001-2000 standards and Technikon's policies and procedures to promote Technikon's continuous improvement.

2. SCOPE

This procedure applies to all Departments within Technikon

3. DEFINITIONS

ISO-9001 Coordinator = ISO-9001 Coordinator or designate
IAFF = Internal Audit Finding Form, Form #XXX (Form located in Technikon Forms Manual)

4. RESPONSIBILITY

4.1. **The ISO-9001 Coordinator** is responsible for assuring that this procedure is followed.

4.2. **The Quality System** is audited internally by people who are independent of the Department under audit and have been certified as internal auditors.

5. PROCEDURE

5.1. **The ISO-9001 Coordinator** establishes a schedule for internal audits at the beginning of each calendar year. This schedule assures that each of the departments is audited against the ISO-9001 standards at least once during the calendar year. The department's areas will be spot-checked for compliance to all the ISO-9001 standards.

5.2. **The ISO-9001 Coordinator** establishes the audit schedule, assigns the Lead Auditor and audit team members, if needed, and notifies them of the audit month and department.

5.3. **Prior to the Audit**, the Lead Auditor shall do the following:

ISO-9001 REFERENCE

5.3.1. Contact the appropriate Department Head to identify a date to perform the audit during the month established by the ISO Coordinator as indicated on the schedule.

5.3.2. Review the previous Non-Conformance Report(s), if applicable.

5.3.3. Review the previous Internal Audit Checklists, if applicable.

5.3.4. Determine what area(s) to audit.

5.3.5. Prepare audit checklist.

NOTE: Areas will be spot checked since every procedure and method cannot be reviewed annually

5.4. **The Lead Auditor** shall meet with the audit team members prior to the audit to discuss the following:

5.4.1. Review of the Internal Audit Checklist and confirmation that the audit team members understand the objectives and the steps in the audit.

5.4.2. If necessary, modification of the Internal Audit Checklist based on discussion with the audit team members.

5.4.3. Assignment of audit responsibilities to all team members.

5.4.4. Review of the importance of note taking on the Internal Audit Checklist and proper completion of Internal Audit Finding Form (IAFF).

5.5. **The Audit Team** conducts an opening meeting with the Head of the Department to be audited and/or designee. The team explains the audit process and what specific areas will be reviewed.

ISO-9001 REFERENCE

5.6. **The Audit Team** shall interview department personnel and review the appropriate department manuals and records to verify compliance with the ISO-9001 standards and Technikon department policies and procedures. Any failures to comply with the ISO-9001 standards or policies and procedures shall be documented on an individual IAFF. The IAFF will indicate a Non Conformance Rating Scale as follows:

5.6.1. **Major** No documented procedure/lack of consistency, major non-conformance with standard.

5.6.2. **Middle** Consistence of practice, but no/vague written procedures; many procedural deviations.

5.6.3. **Minor** Written procedure, but poor document control; minor procedural deviations.

5.7. **The Audit Team** shall meet to review all IAFF's and discuss the audit. Once the audit team is satisfied that they have identified all issues, a close-out meeting shall be held with the Head of the audited Department and/or designee.

5.8. **During the Close-Out Meeting**, all audit findings and comments shall be discussed to insure that all issues have been properly identified. The Department Head or designee will sign each IAFF indicating agreement with the finding. If the Head of the audited Department and/or designee feels a finding is incorrect, the lead auditor and Department Head will discuss the finding and review the applicable standard, policy or procedure.

NOTE: An action plan to address all findings, person(s) responsible and projected completion date(s) will be agreed upon within a week of the audit.

ISO-9001 REFERENCE

5.9. **After the Close-Out Meeting**, the Lead Auditor shall submit the Internal Audit Checklist(s) and IAFF(s) to the ISO-9001 coordinator. Where appropriate, the ISO-9001 Coordinator may recommend voiding an IAFF after review of the applicable procedure, policy and standard, and confirmation of the observation. This review will be conducted for any non-conformances with which the Department Head does not agree. This recommendation must be approved by the Technikon President.

5.10. **The ISO-9001 Coordinator** shall present the IAFF(s) to the Technikon President with a cover letter summarizing the audit. This report is due within 30 days after the audit is conducted.

5.11. **The Technikon President** shall review the audit and approve the recommendations. If the Technikon President does not approve the recommendations, the ISO-9001 Coordinator and appropriate Department Head will revise the recommendations until they are approved.

5.12. **The ISO-9001 Coordinator** shall retain all audit records.

5.13. **The ISO-9001 Coordinator** shall maintain contact with the persons responsible for completing the recommendations. If corrective action has not been completed within one month after the target date for completion, the ISO-9001 Coordinator shall send written notification to the Technikon President and, where applicable, the Supervisor of the person responsible for completing the corrective action. The Technikon President will set a modified target date.

5.14. **The Next Internal Audit Team** shall verify the implementation and effectiveness of corrective action as part of the successive audit conducted on each department.

ISO-9001 REFERENCE

5.15. **<u>The ISO-9001 Coordinator</u>** or designate shall present a summary of the status of the internal audits and the findings at each Management Review Meeting. An Audit Finding Matrix chart will be kept current for the meetings to indicate problem areas.

INTERNAL AUDIT SCHEDULE

DEPARTMENT	**J**	**F**	**M**	**A**	**M**	**J**	**J**	**A**	**S**	**O**	**N**	**D**
Administration		**X**										
Measurement Technology					**X**							
Operations								**X**				
Environmental health and Safety											**X**	

NOTE: Internal audits will mainly be checking for compliance and system continuity. SOP's will only be spot checked.

CPM-1008
ATTACHMENT 1

INTERNAL AUDIT CHECKLIST

DEPARTMENT AUDITED: _____

DATE OF AUDIT _____ **AUDITORS:** _____

_____

_____

APPLICABLE ISO-9001 STANDARD	LOOK AT	LOOK FOR	COMMENTS

CPM-1008
ATTACHMENT 2

ISO-9001 REFERENCE

1. PURPOSE

To ensure that all departures from planned quality standards, including customer complaints, shall be investigated and recorded with a view toward analysis, cause identification, preventive action, and changes to procedures as required.

2. SCOPE

2.1. **Corrective Action** and any other problem areas will be addressed through the following applicable systems:

2.1.1. Equipment Corrective Action
2.1.2. Complaint/Request for Action (CRA) Form #XXX
2.1.3. Suspected Bad Data
2.1.4. Internal Quality Audit
2.1.5. Quality System/Process

2.2. **Preventive Action** will be addressed through the following application systems

2.2.1. Annual Standard Operating Procedure Review
2.2.2. Prescribed interval equipment calibration/verification
2.2.3. Document equipment maintenance and repair
2.2.4. Employee Training
2.2.5. Management Review Meetings

3. DEFINITIONS

SOP = Standard Operating Procedure

ISO-9001 REFERENCE

4. PROCEDURE - CORRECTIVE ACTION

4.1. Equipment Corrective Action System

4.1.1. When quality related equipment is found to be outside its calibration specifications, it will be immediately recalibrated or placed out of service to await recalibration. Applicable calibrations are documented in the equipment calibration/verification log or as specified in Department SOP's. The Department Head, or designate, initiates notification to customers whose data may have been affected by the "out of calibration" equipment if the data has been released. The Critical Procedure entitled "Test Equipment Calibration/Verification & Maintenance", Document # CPM-1010 explains in detail the procedure to be used for "out of calibration" equipment, including the documentation required.

4.2. Complaint/Request for Action System (CRA)

4.2.1. When a customer wishes to register a complaint or initiate a request for some type of action, a CRA form will be filled out (See Forms Manual for form). The form is filled out by a member of the Technikon Department involved.

4.2.2. The ISO-9001 Coordinator, or designate will check the form for completeness to insure the reason for the complaint is clearly identified and documented, a recommendation for corrective action is shown and a responsible person assigned.

4.2.3. Corrective Action to eliminate the cause of the complaint will be initiated. This will be shown in the "action taken" step of the form.

4.2.4. After the CRA has been verified or disclaimed, the form will be completed, the customer will be notified of the findings, if pertinent, and the form will be signed and dated by the responsible person.

ISO-9001 REFERENCE

4.2.5. Once the CRA has been resolved, the ISO 9001 Coordinator or designate will review the CRA, sign it, and date it. The record will be kept in the Technikon CRA file permanently.

4.3. Suspected Bad Data System

4.3.1. When data has been generated that are suspect for any reason, the data will be rechecked for confirmation. If data are confirmed, but still suspect, the test equipment calibration will be verified, if possible, and then the data will be rechecked again. If the equipment is out of calibration, refer to Section 4.1.

4.3.2. If nonconforming data or reports are discovered after distribution, a "Complaint/Request for Action" Form is initiated to resolve the issue. Follow procedure in Section 4.2 of CPM-1009.

4.4. Internal Quality Audit

4.4.1. The ISO 9001 Coordinator is responsible for tracking corrective actions in this system. The IAFF's will be tracked, and the status of corrective actions will be reported to the Technikon President, as part of the quality management system. Refer to "Internal Audit Policy", Document No. CPM-1008 for operation of the internal audit system.

4.5. Quality System/Process

4.5.1. The Quality System and Process for Technikon will be reviewed at the Management Review Meeting, which is held at least annually. At this time, any changes needed to improve the System or process will be discussed and initiated within Technikon.

ISO-9001 REFERENCE

5. PROCEDURE - PREVENTIVE ACTION

5.1. Annual Standard Operating Procedure review

5.1.1. Standard Operating Procedures including Calibration Procedures will be reviewed on an annual basis. Records of those reviews will be maintained within each department. A system to track the review process and to note when the review is due will be maintained by each Technikon Department. By this process, errors in methodology, SOP's and procedures can be corrected.

5.2. Prescribed Internal Equipment Calibration/Verification

5.2.1. Calibration/Verifications of Equipment will be established within each department according to written calibration procedures, and SOPs. These methods will insure systems are working correctly. The frequency will be dictated by the procedures in each department, and records of these data will be maintained within each department.

5.3. Document Equipment Maintenance and Repair

5.3.1. All quality related maintenance and repair work performed on equipment will be documented and a record maintained. This will provide a history on that piece of equipment. Refer to Department Policy & Procedure Manuals for location of documentation.

5.4. Employee Training

5.4.1. All training in methodology will be documented to show employees' proficiency and capability to perform all quality related activities. For additional information on training, See Critical Procedures CPM-1007 entitled Personnel Training.

ISO-9001 REFERENCE

5.5. Management Review Meetings

5.5.1. "Complaint/Request for Action" forms will be discussed at Management Review Meetings to look for common, long term, or repeated problems occurring in the Quality System so they can be addressed and corrected. Any other problems with the Quality System will be resolved at the Management Review Meeting.

6. DOCUMENTATION

6.1. **All Complaint/Request for Action** forms will be reviewed and signed by the ISO Coordinator or designate. The section entitled "Action Taken and Status" will provide closure of the problem, and any follow-up needed will be documented with a date for the follow-up.

6.2. **All Complaint/Request for Action forms and Their Outcome** will be reviewed at the annual Management Review Meeting as per Section 1.3 of the Quality Manual. Records of the meeting will be maintained in the ISO-9001 Library.

6.3. **In Addition**, whenever a specific incidence of quality system or service non-conformance is encountered, investigative protocol shall include an assessment of whether or not the specific failure is indicative of a more general failure having the potential to affect similar services or systems. If so, the corrective action in the specific instance shall be broadly applied in a preventive sense.

TECHNIKON LLC

MANUAL TITLE				Document #
CRITICAL PROCEDURES MANUAL				CPM-1010
DOCUMENT TITLE				ISSUE DATE
EQUIPMENT CALIBRATION/VERIFICATION & MAINTENANCE				
APPROVED BY	PREPARED BY:	PAGE	OF	SUPERCEDES
	C. R. Glowacki	1	4	NEW

ISO-9001 REFERENCE

1. PURPOSE

To assure accurate quantitative results through the regular maintenance and calibration or verification of equipment. Also to handle data generated from equipment that has been found to be out of calibration.

2. SCOPE

This procedure applies to all Technikon equipment.

2.1 **Equipment** that may be calibrated/verified by the user.
2.2 **Equipment** that must be calibrated/verified by an approved contractor.
2.3 **Equipment** that is verified to be outside of specified tolerances.
2.4 **Standards** or Equipment used to Calibrate or Verify Equipment.

3. CONDITIONS

If specific conditions are required, they will be listed in the Department's SOPs and Calibration Procedures.

4. DEFINITIONS

NIST = National Institute of Standards and Testing

5. PROCEDURE

5.1. Equipment Calibrated/Verified by User

5.1.1. All equipment must be inspected and calibrated or verified by a proficient employee before being put into service, and at regular intervals as stated in the Calibration Procedure or SOPs for that department. The procedures and SOPs are found in each Department's Policy and Procedures Manual. These procedures and SOP's will address accuracy, frequency, and where the data are stored.

ISO-9001 REFERENCE

5.1.2. The Calibration Procedure shall conform to the procedure recommended by the Manufacturer or as specified in "Standard Test Methods" or SOPs. When possible, standards will be NIST traceable, or manufacturer's equivalent, and shall be stored in an area so they can be used only for calibration or verification.

5.1.3. A calibration and maintenance record must be kept for every piece of equipment that affects quality, and shall be maintained within each department. The record may be hard copy or electronic. Each record shall contain or reference at least the following information.

5.1.3.1. The identity of the equipment. (Vendor, Serial Number, Model, or any other identification)

5.1.3.2. The date of calibration/verification or maintenance.

5.1.3.3. The results of calibration/verification or maintenance.

5.1.3.4. The date of the standard's last calibration, if required.

5.1.3.5. The initials or signature of the person performing the calibration/verification or maintenance.

5.1.4. If applicable, the unit that has been calibrated/verified or maintained will be tagged with a tag identifying the equipment, the calibration date, tester, and the next calibration date.

5.2. <u>Equipment Calibrated/Verification by Qualified Contractor</u>

5.2.1. All equipment, which due to their complexity or need for highly trained personnel, must be cleaned and calibrated by an approved contractor as stated in the equipment manuals.

5.2.2. If applicable, the unit that has been verified or calibrated, will be tagged with a tag indicating the equipment, its serial number, the calibration date, name of the person conducting the verification or calibration, and the next calibration date, if applicable.

ISO-9001 REFERENCE

5.2.3. Records of work done by Contractors will be maintained by each department, either hard copy or electronic.

5.3. <u>Equipment Out of Specified Tolerances</u>

5.3.1. When a piece of equipment is found to be outside the normal tolerances allowed by the Calibration Procedure or SOP, the unit will be adjusted to within the normal tolerance, if possible. The before and after calibration data is to be recorded if adjustments are made. Each department's Policy and Procedures Manual will address what must be done with a unit out of specification.

5.3.2. If the calibration to the specified tolerance is not possible, the instrument will be removed from service.

5.3.3. The Department Head or designated representative will attempt to determine the actual time the unit was out of tolerance by reviewing available data from the time period between the last and present calibration dates. Customers will be notified their data may be in error.

<u>NOTE:</u> Some protocols or SOP's require that standards or known spikes be run periodically during analyses. If out of specification data is determined, one need only go back to the time that standards gave good data.

5.3.4. When the actual date the unit began reporting inaccurate results has been determined, all tests subsequent to that date will be reevaluated and the corrected data reported.

5.3.5. Customers will be notified of the corrected data.

5.4. <u>Standards or Equipment used to Calibrate or Verify Instruments</u>

5.4.1. Standards or equipment used to calibrate or verify instruments will be addressed in each Department's Policy and Procedures Manual or the Department's SOP's or Calibration Procedures.

ISO-9001 REFERENCE

5.4.2. The standards or equipment used will be listed, the location of the documentation showing authenticity will be indicated, and the frequency of renewing the standard or recalibrating the equipment will be indicated.

6. REFERENCES

6.1. **SOP's and Calibration Procedures in Department Policy and Procedures Manual**.

7. DOCUMENTATION

7.1. **Any Documentation** will be defined and maintained as stated in the SOP's or Calibration Procedures for the Technikon Departments.

TECHNIKON LLC

MANUAL TITLE				Document #
CRITICAL PROCEDURES MANUAL				CPM-1011
DOCUMENT TITLE				ISSUE DATE
PURCHASING				
APPROVED BY	PREPARED BY:	PAGE	OF	SUPERCEDES
	C. R. Glowacki	1	12	

ISO-9001 REFERENCE

1. PURPOSE:

To assure the quality of goods and services purchased by Technikon.

2. SCOPE:

This procedure applies to the procurement of capital equipment, operating supplies, and services.

2.1 **Purchasing Procedures**
2.2 **Procurement of Capital Equipment**
2.3 **Procurement of Operating Supplies**
2.4 **Procurement of Services**
2.5 **Verification of Equipment, Product, or Service Receipt and Quality**

3. REFERENCE:

None

4. DEFINITIONS:

None

5. PROCEDURE:

5.1. Purchasing Procedures - Scope 2.1

5.1.1. Written purchase orders may be completed by any member of the Technikon staff. Each completed purchase order will be reviewed and, if approved, signed by an authorized person including the Technikon President, vice presidents, or controller. The signed purchase order shall be assigned a purchase order number by the controller or designate. The order may be placed with an approved vendor by the controller or designate, or the originator of the purchase order.

ISO-9001 REFERENCE

5.1.2. Each Technikon department will have its own purchasing log where all purchase orders for that department are entered. This log may be kept in hard copy or electronically and contain at least the originator, vendor, date of order, date order received, and whether the order was filled satisfactorily.

5.1.3. Vendor Deficiencies

5.1.3.1. **"Written" Purchase Orders** Any vendor problems involving a "written" purchase order will be handled by the respective Technikon employee. Any unresolved vendor problem and actions taken will be documented on the Vendor Deficiency Form, Form #XXX, and kept in the Vendor Deficiency Records binder located in the ISO library. A copy of this record will be forwarded to the purchase order originator.

5.1.3.2. **"Verbal" Purchase Orders** Verbal purchase orders are not allowed within the Technikon Quality System.

5.2. Procurement of Capital Equipment - Scope 2.2

5.2.1. Vendors of capital equipment may supply Technikon by completing a qualification program prior to approval. The qualification program shall include one (1) or more of the following criteria.

5.2.1.1. ISO certification

5.2.1.2. A history (>3 years) of supplying products acceptable to Technikon.

5.2.1.3. Traceability to a national or consensus standard such as NIST.

5.2.1.4. Passing a performance and/or process audit conducted by a Technikon staff member.

NOTE: A waiver from qualification will be granted with a signed declaration stating the reason for the waiver.

ISO-9001 REFERENCE

5.2.2. A list of approved Capital equipment vendors is attached. These vendors may also be used for additional equipment, services, and/or supplies.

5.2.3. There will be times where capital equipment will be purchased for the purpose of evaluating new and/or experimental analytical techniques, instruments and vendors. It will not be necessary to purchase this equipment from an approved capital equipment vendor. If the results of an analysis using this equipment are provided to customers, then the experimental/evaluative nature of the analyses will be so indicated as part of the report.

5.3. <u>Procurement of Operating Supplies - Scope 2.3</u>

5.3.1. Vendors of operating supplies may supply Technikon by completing a qualification program prior to approval. The qualification program shall include one (1) or more of the following criteria.

5.3.1.1. ISO certification

5.3.1.2. A history (>3 years) of supplying products acceptable to Technikon.

5.3.1.3. Traceability to a national or consensus standard such as NIST.

5.3.1.4. Passing a performance and/or process audit conducted by a Technikon staff member.

<u>NOTE:</u> A waiver from qualification will be granted with a signed declaration stating the reason for the waiver.

5.3.2. A list of approved operating supplies vendors is attached. These vendors may also be used for additional equipment, services, and/or supplies.

ISO-9001 REFERENCE

There will be times where operating supplies will be purchased for the purpose of evaluating new suppliers and vendors. It will not be necessary to purchase these supplies from an approved operating supplies vendor.

Publishers of reference books, encyclopedia, etc., are not required to be on the list of approved operating supplies vendors.

5.4. Procurement of Services

5.4.1. Vendors of services may supply Technikon by completing a qualification program prior to approval. The qualification program shall include one (1) or more of the following criteria

5.4.1.1. ISO certification

5.4.1.2. A history (>3 years) of supplying services acceptable to Technikon.

5.4.1.3. Traceability to a national or consensus standard such as NIST.

5.4.1.4. Passing a performance and/or process audit conducted by a Technikon staff member.

NOTE: A waiver from qualification will be granted with a signed declaration stating the reason for the waiver.

5.4.2. A list of approved service vendors is attached. These vendors may also be used for additional equipment, services, and/or supplies. There will be times where services will be purchased for the purpose of evaluating new service providers. It will not be necessary to purchase these services from an approved service vendor.

ISO-9001 REFERENCE

5.5. Verification of Equipment, Product, or Service Receipt and Quality - Scope 2.5

5.5.1. Verification of the receipt of capital equipment, operating supplies and services is the responsibility of the person initiating the purchase requisition. Verification can also be conducted by a co-worker who is familiar with the use of the purchased equipment, product, or service. The receipt of material or services is documented by one or more of the following:

5.5.1.1. Completion of a “receipt of materials”.

5.5.1.2. Completion of the Department Purchasing log (either electronically or hard copy) Refer to 5.1.2 this procedure.

5.5.2. Verification of the quality of capital equipment, operating supplies, and services is the responsibility of the person initiating the purchase requisition. Verification can also be conducted by a co-worker who is familiar with the use of the purchased equipment, product, or service. Verification criteria may include:

5.5.2.1. Equipment or services meet or exceed published Vendor specifications.

5.5.2.2. Certificate of analysis.

5.5.2.3. Label purity certification i.e. Analytical Reagent Grade, USP, etc.

5.5.2.4. Other criteria acceptable to the responsible person.

5.5.3. The acceptance of materials or services is documented by one or more of the following:

5.5.3.1. Approving the vendor’s invoice for payment.

ISO-9001 REFERENCE

5.5.3.2. Allowing payment of invoices for materials with a "receipt of materials".

5.5.3.3. Indicating "yes" in the Department Purchasing Log under "Acceptable".

6. DOCUMENTATION:

6.1. Each Department within Technikon maintains a hard copy or electronic copy of all initiated orders.

6.2. Invoices coded and approved by Technikon are maintained in the Technikon Administration Department.

APPROVED VENDOR LIST - CAPITAL EQUIPMENT

VENDOR	EQUIPMENT
Agilent	Chromatograph Accessories
Agilent	Chromatographic Equipment
Agilent	Electronic
Alltech	Chromatograph Accessories
Balston	Chromatograph Accessories
California Instruments	On-Line Monitors
CDS	Chromatograph Accessories
ChromTech	Chromatograph Accessories
Environics	Chromatograph Accessories
Fisher Scientific	Chromatographic Equipment
hNU	Chromatograph Accessories
ISCO	Chromatograph Accessories
ISCO	Chromatographic Equipment
JM Science	Columns
Leap Technologies	Chromatograph Accessories
Mettler	Balances
Millipore	Water Purification Systems
National Instruments	Computer/Network Equipment
Redford Core Blowers	Core Making Equipment
Sartorious	Balances
Tween-Albert	Materials Testing Equipment
Varian	Vacuum Leak Detector
Varian Instruments	Chromatograph Accessories
Varian Instruments	Chromatographic Equipment

APPROVED VENDOR LIST - CAPITAL EQUIPMENT

VENDOR	EQUIPMENT
VIG Instruments	On-Line Monitors
Whatman	Chromatograph Accessories

APPROVED VENDOR LIST - OPERATING SUPPLIES

VENDOR	EQUIPMENT
3M	Laboratory Supplies
Ace Glass	Laboratory Supplies
Adward	Laboratory Supplies
Aeltec	Laboratory Supplies
Air Product	Laboratory Supplies
Aldrich Chemical	Laboratory Supplies
Aldrich Chemical Company	NMR Solvents
Allied Electronics	Electronics Supplies
All-Pack, Inc.	Laboratory Supplies
Alltech	Chromatographic Supplies
Altex	Chromatographic Supplies
American Air Gases	Laboratory Supplies
Chemical Research Supplies	Chromatographic Supplies
Chrom Tech	Chromatographic Supplies
Chrompack	Chromatographic Supplies
Cole Parmer	Laboratory Supplies
Denver Instruments	Calibration Standards
Fisher Scientific	Laboratory Supplies
Fluka Chemical Corp.	Specialty Chemicals
Gelman Sciences	Filtration Equipment
Hamilton	Chromatographic Supplies
Harris Gas	Compressed and Liquefied Gases
IOTECH	Electronics Supplies
ISCO	Chromatographic Supplies

APPROVED VENDOR LIST - OPERATING SUPPLIES

VENDOR	EQUIPMENT
J&W Scientific	GC Columns
Jones Chromatography	Laboratory Supplies
Lancaster Chemicals	Specialty Chemicals
Mallinckrodt Baker	Laboratory Supplies
Matheson	Laboratory Supplies, Compressed Gases
Millipore	Laboratory Supplies
MVAK Technologies Inc.	Laboratory Supplies
National Instruments	Computer/Network Supplies or Software
Newark Electronic	Electronics Supplies
NIST	Laboratory Supplies
Omega Engineering	Laboratory Supplies
Pfaltz & Bauer	Laboratory Supplies
Phenomenex	Chromatographic Supplies
Pierce	Laboratory Supplies
Praxair, Inc.	Compressed Gases
Restek Corp.	Columns, Standards
Scott Specialty Gases	Laboratory Supplies
SGE, Inc.	Syringes
SGE, Incorporated.	Chromatographic Supplies
Sigma Chemical	Chromatographic Supplies
SKC	Laboratory Supplies
Supelco	Chromatographic Supplies
Technical Heaters	Laboratory Supplies
Thermo Environmental	Instrument Supplies
VWR Scientific	Laboratory Supplies

APPROVED VENDOR LIST - OPERATING SUPPLIES

VENDOR	EQUIPMENT
See Capital Equipment List	

APPROVED VENDOR LIST - SERVICES

VENDOR	SERVICE
Clayton Environmental Consultants, Novi, MI division	Laboratory Services
STL Laboratories	Laboratory Services
See Capital Equipment list	
See Operating Supply List	

TECHNIKON LLC

MANUAL TITLE			Document #
CRITICAL PROCEDURES MANUAL			CPM-1012
DOCUMENT TITLE			ISSUE DATE
LOCAL AREA NETWORK - OPERATION			
APPROVED BY	PREPARED BY:	PAGE OF	SUPERCEDES
	C. R. Glowacki	1 1	

ISO-9001 REFERENCE

1. PURPOSE

To define the manner in which Technikon manages its local area network and computer resources

2. SCOPE

This procedure applies to the Technikon Local Area Network and computer resources.

- 2.1 Apparatus
- 2.2 Backup Procedure
- 2.3 Restoration Procedure
- 2.4 References

3 Standard Operating Procedure (SOP) Location

3.1 All of the SOPs associated with the operation and maintenance of the Technikon Local Area network and Computer Resources are located in the Administration Department Tier 3 documents. See AD-XXXX through AD-YYYY.

TECHNIKON LLC

MANUAL TITLE			Document #
CRITICAL PROCEDURES MANUAL			CPM-1013
DOCUMENT TITLE			ISSUE DATE
REPORTING PROCESS			
APPROVED BY	PREPARED BY: C. R. Glowacki	PAGE 1 OF 4	SUPERCEDES

ISO-9001 REFERENCE

1. PURPOSE

To define the manner in which Technikon transmits written information and data to the customer.

2. SCOPE

This procedure applies to reports being issued by Technikon.

2.1 Emissions Test Reports
2.2 Process Development Reports
2.3 CERP Monthly Reports
2.4 Specialty Reports
2.5 Commercial Test Report

3. DEFINITIONS

SOP = Standard Operating Procedures
CERP = Casting Emission Reduction Program
DoD = Department of Defense

4. PROCEDURE

4.1. Emission Test Reports – Scope 2.1

4.1.1. The assigned author of the report collects raw and reduced data, results, and observations from the Measurement Technologies and Production Team members that are involved in a test.

4.1.2. The author drafts a preliminary report for review by the Production and Measurement Technologies teams.

4.1.3. Following revision of the draft, if any, the report is reviewed by the Vice President – Measurement Technologies and the Vice President – Operations.

ISO-9001 REFERENCE

4.1.4. Comments are incorporated into a final report that is distributed after approval and signature by the author, Vice President – Measurement Technologies, the Vice President – Operations, Technikon President, and other appropriate parties.

4.2. Process Development Report – Scope 2.2

4.2.1. The assigned author of the report collects raw and reduced data, results, and observations from the Measurement Technologies and Production Team members that are involved in a test.

4.2.2. The author drafts a preliminary report for review by the Production and Measurement Technologies teams.

4.2.3. Following revision of the draft, if any, the report is reviewed by the Vice President – Measurement Technologies and the Vice President – Operations.

4.2.4. Comments are incorporated into a final report that is distributed after approval and signature by the author, Vice President – Measurement Technologies, the Vice President – Operations, Technikon President, and other appropriate parties.

4.3. Monthly Report – Scope 2.3

4.3.1. The Technikon CERP Program Manager drafts a preliminary monthly report based on information received from the Administrative, Operations, and Measurement Technologies departments.

4.3.2. The draft report is reviewed by the Vice President – Measurement Technologies, the Vice President – Operations, and the Controller.

4.3.3. Comments are incorporated into a final report.

ISO-9001 REFERENCE

4.3.4. The final report is submitted to the DoD CERP Program Manager.

4.4. Specialty Report – Scope 2.4

4.4.1. The assigned author of the report collects raw and reduced data, results, observations, and other pertinent information from the Measurement Technologies Team, the Production Team, and other sources, as needed.

4.4.2. The author drafts a preliminary report for review by the Production and Measurement Technologies teams.

4.4.3. Following revision of the draft, if any, the report is reviewed by the Vice President – Measurement Technologies and the Vice President – Operations.

4.4.4. Comments are incorporated into a final report that is distributed after approval and signature by the author, Vice President – Measurement Technologies, the Vice President – Operations, Technikon President, and other appropriate parties.

4.5. Commercial Test Report- Scope 2.5

4.5.1. The assigned author of the report collects raw and reduced data, results, and observations from the Measurement Technologies and Production Team members that are involved in a test.

4.5.2. The author drafts a preliminary report for review by the Production and Measurement Technologies teams.

4.5.3. Following revision of the draft, if any, the report is reviewed by the Vice President – Measurement Technologies and the Vice President – Operations.

4.5.4. Comments are incorporated into a final draft report that is distributed to the client for review and approval.

ISO-9001 REFERENCE

4.5.5. Client comments, if any, are incorporated into the final report and the report is approved and signed by the author, Vice President – Measurement Technologies, the Vice President – Operations, Technikon President.

4.5.6. The signed report is distributed to the client.

TECHNIKON LLC

MANUAL TITLE			Document #
CRITICAL PROCEDURES MANUAL			CPM-1014
DOCUMENT TITLE			ISSUE DATE
DESIGN AND/OR DEVELOPMENT CONTROL			
APPROVED BY	PREPARED BY: C. R. Glowacki	PAGE 1 OF 4	SUPERCEDES

ISO-9001 REFERENCE

1. PURPOSE

To define the procedures used by Technikon when implementing, documenting, verifying, and controlling design and development functions associated with non-routine testing and process improvement.

2. SCOPE:

Applicable to the following Design and Development Functions.

2.1 Emission Testing
2.2 Process Development
2.3 Commercial Activities

3. DEFINITIONS:

None

4. PROCEDURE

4.1. Emission Testing – Scope 2.1

4.1.1. When a product is identified for non-routine testing under the CERP program a Test Approval Form is (Form #XXX) completed by a member of the Technikon Quality System.

4.1.2. The draft Test Approval Form is reviewed by the Vice President – Operations and the Vice President – Measurement Technologies and signed to document approval.

4.1.3. The signed form is sent to the Manager – Process Engineering who drafts a Technikon Test Plan (Form #XXX).

ISO-9001 REFERENCE

4.1.4. The draft Test Plan is reviewed by the Vice President – Operations and the Vice President – Measurement Technologies and signed to document approval and returned to the Manager – Process Engineering with a copy going to the Manager – Laboratory Services.

4.1.5. The Manager – Process Engineering or designate prepares Process Instructions for the test. The Process Instructions are reviewed by the Vice President – Operations.

4.1.6. The Manager – Laboratory Services or designate prepares a Test Sampling Plan for the test. The Test Sampling Plan is reviewed by the Vice President – Measurement Technologies.

4.1.7. Following completion of the test a report is prepared. See CPM-1013.

4.2. Process Development – Scope 2.2

4.2.1. When a process is identified for non-routine development and/or testing under the CERP program a Test Approval Form is (Form #XXX) completed by a member of the Technikon Quality System.

4.2.2. The draft Test Approval Form is reviewed by the Vice President – Operations and the Vice President – Measurement Technologies, if the process evaluation includes emission testing, and signed to document approval.

4.2.3. The signed form is sent to the Manager – Process Engineering who drafts a Technikon Test Plan (Form #XXX).

ISO-9001 REFERENCE

4.2.4. The draft Test Plan is reviewed by the Vice President – Operations and the Vice President – Measurement Technologies, if the Test Plan includes emission testing, and signed to document approval and returned to the Manager – Process Engineering with a copy going to the Manager – Laboratory Services, if emission testing is included in the plan.

4.2.5. The Manager – Process Engineering or designate prepares Process Instructions for the test. The Process Instructions are reviewed by the Vice President – Operations.

4.2.6. The Manager – Laboratory Services or designate prepares a Test Sampling Plan for the test, if emission testing is included in the test plan. The Test Sampling Plan is reviewed by the Vice President – Measurement Technologies.

4.2.7. Following completion of the test a report is prepared. See CPM-1013.

4.3. Commercial Activities – Scope 2.3

4.3.1. When a client contacts Technikon to conduct a non-routine commercial test and/or development activity, the Vice President – Measurement Technologies or the Vice President – Operations gathers the pertinent information needed to define the test and prepares a proposal including projected costing.

4.3.2. The draft proposal is submitted to the client for comment and/or approval.

4.3.3. Comments are included in a revised proposal that is submitted to the client for approval.

4.3.4. Work on the proposed test does not begin before a purchase order is received from the client.

ISO-9001 REFERENCE

4.3.5. The draft Technikon Test Plan (Form #XXX) is prepared by the Manager – Process Management.

4.3.6. The draft Test Plan is reviewed by the Vice President – Operations and the Vice President – Measurement Technologies, if the Test Plan includes emission testing, and signed to document approval and returned to the Manager – Process Engineering with a copy going to the Manager – Laboratory Services, if emission testing is included in the plan.

4.3.7. The Manager – Process Engineering or designate prepares Process Instructions for the test. The Process Instructions are reviewed by the Vice President – Operations.

4.3.8. The Manager – Laboratory Services or designate prepares a Test Sampling Plan for the test, if emission testing is included in the test plan. The Test Sampling Plan is reviewed by the Vice President – Measurement Technologies..

4.3.9. .Following completion of the test a report is prepared. See CPM-1013.

TECHNIKON LLC

MANUAL TITLE	
CRITICAL PROCEDURES MANUAL	
DOCUMENT TITLE	
ATTACHMENT 1	

Application Method

DOCUMENT NO.	
MT-6000	
SAMPLE TRAIN CALIBRATION AND USE	PAGE 1 OF 3

DATE ISSUED	SUPERSEDES	PREPARED BY
	New	C. Hornsby
APPROVED BY	APPROVED BY	APPROVED BY

1. SCOPE

1.1. The method is applicable for the calibration and use of the sampling trains used by Technikon, LLC. The method can also be utilized for other similar air emissions sampling applications as needed.

2. SUMMARY OF METHOD

1.1. Samples are collected using critical orifice technology under one vacuum system from a sampling probe located at the centroid of the duct. Sampling parameters are derived from US EPA, NIOSH and OSHA methods.

3. MATERIALS

1.2. Critical orifices, various sizes

1.3. Balston air filters or equivalent

1.4. Temperature probe

1.5. Heated sampling manifold – Technikon design

1.6. Technikon critical orifice sampling train

1.7. Pressure gauge – readability to 0.5 inches Hg

1.8. Rotary vane vacuum pump

1.9. *Magnahelic* pressure differential meter

1.10. Silicone tubing – various sizes as needed

1.11. *Teflon* tubing - various sizes as needed

1.12. *Tygon* tubing - various sizes as needed

1.13. Bios Drycal flowmeters – range from 10mL/min to 50 L/min

1.14. Sample media (various types)

1.15. Silica Gel

4. PROCEDURE

1.16. Sample Train Calibration

1.16.1. Based on the Test Sample Plan, attach appropriate critical orifices and "bodies" to designated channels. Check all fittings for leaks.

1.16.2. Turn on vacuum and wait until pressure the gauge reads greater than 19 in. Hg. Using the *Bios Drycal* flowmeters, check the flows of each critical orifice with the appropriate media in-line. Record results on the Organic Train Calibration Sheet. If all flows are within prescribed parameters, close the vacuum valve. See Appendix A for Organic Train Calibration Sheet example.

1.17. Sample Train Set-up and Testing Environment Parameters

1.17.1. Measure the ambient temperature near the sampling train. Record the ambient temperature on the Organic Train Calibration Sheet.

1.17.2. Insert the heated manifold into the sampling port. Check the temperature of the manifold to be sure it is above 120°C.

1.17.3. Attach all labeled sample media onto sampling train according to the Test Sample Plan. Check the direction of flow for all media types and align them vertically.

1.17.4. Once the Process team is ready to begin the test (i.e. pour the molten metal), turn on the vacuum of the sampling train. Watch until the pressure reaches at least 19 in. Hg and signal the start of the pour. Record other data as required by the Organic Train Calibration Sheet. Note all problems and observations on the data sheet.

1.17.5. At the termination of the test run, turn off vacuum and wait for the pressure to drop to zero. Cap all sampling media and seal them in a zip lock bag. Store at 4 ± 2ºC until analyzed.

2. Appendix A

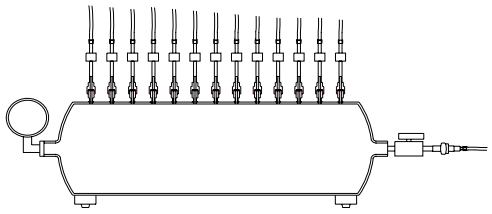
TECHNIKON – ORGANIC TRAIN CALIBRATION SHEET

GENERAL TEST INFORMATION

DATE:	4/28/2005			
FOUNDRY NAME:	Research			
STACK NAME:	Total	**DIAMETER:**	5.834	
SERIES:	GQ	**SAMPLING Run #:**	Pre-test cal	
SAMPLE PORT:		**DISTANCE IN:**		
SAMPLER:	Girocco			
TRAIN ID:	TECHNIKON-1	**TEST TIME:**	75	*min*

GENERAL TEST PARAMETERS

BAROMETRIC PRESSURE *(in. Hg)* **:**		
STATIC PRESSURE *(in. H_2O)*:		(Always Negative)
STACK TEMP AVG *(°F)* **:**		
AMBIENT TEMP *(°F)* :		
TRAIN VACUUM *(in Hg)* **:**	>20	
REGULATED VACUUM *(in Hg)* **:**	23-25	
UNREGULATED VACUUM *(in Hg)* **:**	>25	



	Vacuum	Ambient	Time
OPEN	23.0	64.9	10:52
MEDIA	23.0	64.2	11:14

EQUIPMENT CALIBRATION

Channel	Target *ml/min*	Serial #	Size *microns*	Media	Type	OPEN	MEDIA	Average	% Diff.	Comments
1	*30*	30-1-31.31-N	N/A	M-18	S	31.22	31.18	31.20	*-0.128*	
2	*30*	30-5-31.44-R	N/A	M-18	S	31.28	31.29	31.29	*0.032*	
3	*30*	30-6-31.56-R	N/A	M-18	S	31.25	31.26	31.26	*0.032*	
4	*60*	60-1-62.01-N	N/A	Excess	N/A			N/A	*N/A*	
5	*500*	400-13-481.5-N	N/A	OSHA ID200	S	484.4	479.3	481.9	*-1.053*	
6	*500*	400-14-481.0-N	N/A	OSHA ID200	S	482.5	477.9	480.2	*-0.953*	
7	*1000*	1000-1-1030-N	N/A	NIOSH 1500	S	1023	981.1	1002.1	*-4.096*	
8	*1000*	1000-2-1036-N	N/A	NIOSH 1500	S	1036	993.9	1015.0	*-4.064*	
9	*1000*	1000-5-1148-N	N/A	TO11	S	1148	1003.0	1075.5	*-12.631*	
10	*1000*	1000-6-1179-N	N/A	TO11	S	1177	1018.0	1097.5	*-13.509*	
11	*1000*	1000-3-1033-N	N/A	Excess	N/A			N/A	*N/A*	
12	*500*	400-12-492.3-N	N/A	Moisture	S	493.0	493.8	493.4	*0.162*	
13	*5000*	5000-1-5099-N	N/A	Excess	O	5099	5144	5122	*0.883*	

Comments:

3.

Application Method

DOCUMENT NO.	
MT-6001	
BALANCE CALIBRATION AND VERIFICATION	PAGE 1 OF 2

DATE ISSUED	SUPERSEDES	PREPARED BY
	New	C. Hornsby
APPROVED BY	APPROVED BY	APPROVED BY

1. SCOPE

The method is applicable for the calibration and verification of the following balances and scales maintained at Technikon, LLC.

Scale Model	Capacity	Readability	Location	Serial Number
Mettler Analytic (Denver) XE-100	100g	0.1 mg	Sand Lab	
Mettler Platform PB302	310g	0.2 g	Sand Lab	
Mettler Platform PJ4000	4000g	0.1 g	PCS Area	J21438
Mettler Platform PB8000	50-8000g	1.0 g	Sand Mixing	2115215118
Ohaus Platform MP-2 (OS-10)	110 Lbs	0.05 Lbs	PCS Area	
Cardinal Platform 748-E	2000Lbs	0.5 Lbs	PCS Area	9511-70
Mettler Analytic AG204	210g	0.1 mg	Anal. Lab	1118230092
Westweigh WP1000-10	1000 Lbs	0.5 Lbs	PCS Area	13086-9

2. SUMMARY OF METHOD

3.1. All balances are to be calibrated with National Institute of Standards and Technology (NIST) traceable weights and continuing calibration verifications are to be performed prior to each day's use or at least twice yearly. Calibration verification will be performed any time balances are moved to another location by a proficient Technikon staff member.

3. MATERIALS

3.2. Weights used for calibrations performed:

3.2.1. ASTM Class 1 and Class 2 analytical weights ranging from 5000 g to 0.0001 g.

3.2.2. Pound weight sets with readabilities ranging from 5 Lb to 1 Lb.

3.2.3. Weight certifications will be maintained in the *Equipment Calibration and Certification* binder located in the Technikon Analytical Laboratory.

4. PROCEDURE

3.3. Calibration performed by external firm.

3.3.1. Twice yearly, an outside contractor will perform calibrations on all balances listed above using NIST traceable weights.

3.3.2. Documentation of all problems and/or alterations to balances during calibration will be performed, and copies will be kept in the *Equipment Calibration and Certification* binder.

3.4. Calibration verifications performed internally.

3.4.1. If balance is moved to another location, calibration verification with NIST traceable weights must be performed and documented in the *Equipment Calibration and Certification* binder.

3.5. Other maintenance

3.5.1. Balances will be kept free of debris (e.g. solvents, solid materials) on and around the balance pans. Any residue that cannot be removed by using solvents, soap, or steel wool may remain on the balance.

3.5.2. Check the bubble level/location (if applicable) to see if the balance is level. If the balance is not level, adjust the legs so that the bubble indicator moves into the circle. Once leveled, tare the balance to zero.

3.5.3. Refer to manufacturer instruction manuals for information on operation and warranty.

Application Method

DOCUMENT NO.	
MT-7001	
DOCUMENT TITLE	PAGE OF
DETERMINATION OF ALDEHYDES AND KETONES IN AIR	1 6
DATE ISSUED	SUPERSEDES: NEW
PREPARED BY: C. Hornsby	
APPROVED BY	APPROVED BY
APPROVED BY	

1. SCOPE AND APPLICATION

1.1. This method is applicable for the determination of the following aldehydes and ketones in air:

Analyte	Limit of Detection (μg)
Formaldehyde	0.3
Acetaldehyde	0.3
Acetone	0.3
Acrolein	0.3
Propionaldehyde	0.3
Crotonaldehyde	0.3
2-Butanone	0.3
Butyraldehyde/Methacrolein (co-elution)	0.5
Benzaldehyde	0.3
Valeraldehyde	0.3
o,m,p-Tolualdehyde (co-elution)	0.8
Hexaldehyde	0.3

2. SUMMARY OF METHOD

2.1. A known volume of air is drawn through DNPH treated silica gel sampling tubes to collect aldehyde or ketone vapors. Acetonitrile is eluted through the cartridge and the eluent is adjusted to an appropriate final volume, e.g., 5.0 mL. An aliquot of this solution is injected onto a High Performance Liquid Chromatograph (HPLC). Peak responses are determined and compared with calibration curves obtained from injections of standard solutions.

3. INTERFERENCES

3.1. Acetone is a significant interference with this method. All glassware and other analytical supplies must be entirely free of acetone and the analyst must ensure this analysis is run in an acetone free environment.

3.2. The other only other significant interferences in the method are certain isometric aldehydes or ketones that may be unresolved by the HPLC system. Such interferences can often be overcome by altering the separation conditions (e.g. using alternate HPLC columns or mobile phase compositions).

4. APPARATUS AND MATERIALS

4.1. HPLC system.

4.1.1. Gradient HPLC pump

4.1.2. UV Detector, variable wavelength

4.1.3. Autosampler

4.1.3.1. HPLC autosampler vials

4.1.4. Deltabond-AK 150 mm x 250 mm o.d. column or equivalent

4.2. PC equipped with software for peak integration

4.2.1. DNPH silica gel cartridges (SKC 226-119 or equivalent),

4.2.2. 10 µL syringes

4.2.3. Glass syringes: 10, 25, 100 µL

4.2.4. Pipette: 2 mL capacity

4.2.5. 10 mL graduated cylinders

4.2.6. 14 mL amber vial with polyethylene septum and screw top

5. REAGENTS

5.1. Acetonitrile , HPLC grade

5.2. DNPH derivatives certified to be 99+% pure

5.3. Reagent water, 18 MOhm-cm resistance

5.4. Mobile phase, acetonitrile and reagent water

6. HEALTH AND SAFETY

6.1. Refer to the Material and Safety Data Sheets (MSDS) for information on the toxicity and potential hazards of solvents/chemicals used during this procedure.

7. QUALITY CONTROL

7.1. A laboratory blank (sample media and desorption solvent) is prepared and analyzed with each analytical batch of 20 or fewer samples.

7.2. A field blank should be submitted by the client, and analyzed with each group of 20 or fewer samples. The field blank is treated identically to the samples. The sample performance criteria must be met for field blanks.

7.3. Two recovery samples spiked at a mid-level (1-5 μg) are prepared and analyzed with each analytical batch of 20 or fewer samples.

7.4. A continuing calibration verification standard at mid-level concentration is analyzed at least after every 10th injection, and must be within ±10% of the initial calibration.

8. PROCEDURE

8.1. Preparation of Linear Standards – The linear curve of standards is used for calibration purposes. The standard solutions are made by diluting specific amounts of the bulk standard (containing all the compounds listed in Section 1.1) with acetonitrile. The concentrations of the calibration standards should encompass the expected sample analyte range. A minimum of five (5) concentrations will be used in each curve.

8.2. The calibration solutions must be analyzed under the same liquid chromatographic conditions and during the same period as the unknown samples. This will minimize the

effect of day-to-day variations of the detector response or the composition of the mobile phase.

8.3. To prepare recovery samples, spike 20 μL-100 μL of an aldehyde and ketone standard at upper end of calibration limit onto the DNPH silica gel cartridges. Place caps back on the cartridges and allow 30 minutes to pass before eluting.

8.4. To prepare the laboratory blank, another unused cartridge is treated in the same manner as in Section 8.3, except that neither aldehydes or ketones are added to the media.

8.5. Preparation of Samples

8.5.1. Transfer the front and back sections of DNPH media from the sampled tube into separate vials.

8.6. Desorption

8.6.1. Add a known volume, e.g., 1.0 to 5.0 ml, of acetonitrile into the vial and cap tightly. Allow to stand for at least sixty (60) minutes with occasional shaking.

8.7. Liquid Chromatography Conditions

8.7.1. Mobile Phase: See Appendix A for suggested conditions used successfully.

8.8. Transfer an aliquot of each sample, including quality control samples, into autosampler vials, if an Autosampler is being used.

8.9. Peak Measurement

8.9.1. Measure the response of the sample peak manually or with an electronic integrator. Calculate the amount of aldehydes or ketones from a standard curve prepared as described in Section 8.1.2. See Appendix A for an example of a typical chromatogram.

9. CALCULATIONS

9.1. Results are calculated from a linear regression calibration curve. Calculate the mass (μg) corresponding to the peak response for the particular sample. No volume corrections are needed because the curve is based on μg extracted and the volume of sample injected into the HPLC is identical to the volume of the standards injected.

9.2. In the event that the client blank produces a peak at the same retention times as an aldehyde or ketone peak, the amount of aldehyde or ketone calculated for the blank must be subtracted from the amount calculated for each sample.

9.3. Calculate the average recovery of the two spiked samples:

9.4. $$\% \text{ Recovery} = \frac{\text{Total Amount of Analyte Recovered}}{\text{Total Amount of Analyte Spiked}} \times 100\%$$

9.5. Results are reported in μg.

10. LABORATORY DATA VALIDATION

10.1. See Technikon SOP: XXXXX for data review procedures.

11. REAGENT WASTE DISPOSAL

11.1. Waste is disposed of in accordance with state, county, and federal EPA regulations.

12. REFERENCES

12.1. The Bureau of National Affairs, Inc., Federal Register, Environmental Reporter, Vol. 2, EPA Method TO-11, Washington, D.C., 1996.

Appendix A

Liquid Chromatography Conditions:

Mobile phase: 65/35 water/ACN for 7 min., followed by a gradient to 35/65 over 5 minutes. Hold 35/65 composition until the last compound has eluted and bring back to initial conditions.

Flow rate: 1.5 mL/min.
Sample size: 10-15 μL
UV Detector: 360 nm
Retention Time: see attached chromatogram
Runtime: 15-20 minutes

Application Method

DOCUMENT NO.	
MT-7002	
DOCUMENT TITLE: DETERMINATION OF ALIPHATIC AMINES IN AIR BY GC/NPD	PAGE 1 OF 8
DATE ISSUED	SUPERSEDES: NEW
PREPARED BY: C. Hornsby	
APPROVED BY	APPROVED BY
APPROVED BY	

1. SCOPE

This method is applicable for the determination of the following aliphatic amines:

Dimethylethylamine (DMEA)	Triethylamine (TEA)

2. SUMMARY OF METHOD

2.1. The analytes are collected on silica gel tubes. The media is solvent desorbed and analyzed by Gas Chromatography with a Nitrogen-Phosphorous Detector (GC/NPD). Quantitation is performed by comparison of a calibration curve of known analytes concentrations to the samples (peak area vs. μg analyte).

3. INTERFERENCES

3.1. There are no known interferences.

4. APPARATUS AND MATERIALS

4.1. Gas chromatograph, equipped with a nitrogen-phosphorous detector (NPD), integrator, and column.

4.2. Column: Chromosorb 103 or equivalent

4.3. Autosampler vials, e.g., 1.5 mL, with PTFE-lined crimp caps

4.4. Autopipette – able to dispense 1.0 mL, 1.5 mL, and 2.0 mL

4.5. Personal computer (PC) with chromatography software

4.6. Syringes

4.7. Volumetric flasks –with Teflon stopper

4.8. Class A Volumetric pipettes.

4.9. Vials for desorption

5. REAGENTS

5.1. Methanol (MeOH), GC grade

5.1.1. Desorbing solution: 4:1 MeOH/0.1 N H_2SO_4 aqueous

5.2. Neat Materials: dimethylethylamine and triethylamine; reagent grade, purchased as certified materials.

5.3. Alkalyzing solution: e.g., 0.2 N NaOH

5.4. Gases: nitrogen, helium, hydrogen, and air; GC grade.

6. SAMPLE SHIPMENT, PRESERVATION, AND STORAGE.

6.1. Following sample collection, the ends of the tubes are capped with the caps provided. Samples are shipped to the laboratory with ice.

6.2. Samples are stored in the laboratory in a refrigerator at 4°C and analyzed as soon as possible.

7. HEALTH AND SAFETY

7.1. Refer to the Material Safety Data Sheets (MSDS) for information on toxicity and potential hazards of solvents/chemicals used during this procedure.

8. QUALITY CONTROL

8.1. The desorption efficiencies determine the measure of accuracy of the analytical procedure by comparing known concentrations against percent recovered.

$$\% \text{ recovery} = \frac{\mu g \text{ recovered}}{\mu g \text{ spiked}} \times 100\%$$

Desorption efficiencies are determined for each combination of analytes and sampling media. The initial desorption efficiency study consists of duplicate spikes at three levels corresponding to a low, mid-range, and high calibration level. Duplicate spikes are prepared at one of the calibration levels, with each batch for subsequent analyses.

8.2. Prepare standard solutions.

8.2.1. Add known amounts of specific analyte stock solution (equivalent to the range of samples) to appropriate solvent in 10 mL volumetric flasks and dilute to the mark.

8.2.2. Analyze with samples and media blanks for qualitative identification of derivative peaks.

8.3. Store all spiked media in a refrigerator and allow 12 hours for adsorption.

8.4. Other levels may be spiked if necessary. For example, when the concentration of analytes in the samples falls in a range where no DE data is available.

8.5. Prepare one laboratory media blank for each media type according to Section 10.

8.6. A verification standard must be also prepared from the same neat materials as used for the working standard. The verification standard and the working standard must be linear within ± 10%. See section 10.2 for preparation instructions.

8.7. A continuing calibration STD will be run very 10 injections. The response of the CCVs must be within ± 10%. If a CCV falls out of the range, the samples that are positive bracketed between the CCV must be rerun.

9. PROCEDURE

9.1. Stock Standard preparation

9.1.1. The primary calibration standard is 5 µL of neat material in 10 mL of solvent (5 µL/10mL).

9.1.2. Fill an appropriate, e.g., 10 mL, volumetric flask to approximately 9 mL with the desired solvent using a disposable pipette.

9.1.3. Using a syringe, add 5 μL of neat analyte(s) to the volumetric flask.

9.1.3.1. Rinse the syringe at least 4 times with the neat material.

9.1.3.2. After drawing back past the 5 μL mark, position the syringe upside-down, making sure there are no trapped air bubbles.

9.1.3.3. Carefully push the plunger to the 5 μL mark, wiping off all excess with a Kimwipe before adding to the flask.

9.1.3.4. Take care to rinse the syringe with solvent between analytes.

9.1.3.5. Use a disposable pipette to fill the volumetric flask to the correct dilution mark, e.g., 10 mL.

9.1.3.6. Cap and invert several times.

9.1.4. Dilutions are made from this primary (working) standard. A five-point calibration curve, along with a continuing calibration standard, is prepared.

9.1.4.1. Standard dilutions are made in 10 mL aliquots. Pipette 8 mL of desorbing solution into each volumetric flask. Using a Hamilton syringe, add the appropriate volume of the working standard to each vial for the chosen dilutions as stated below. Bring to a final volume of 10 mL with desorbing solution.

1/1000 dilution = 1μL/1 mL of working standard in 1 mL of solvent
1/500 dilution = 2 μL/1 mL of working standard in 1 mL of solvent.
1/200 dilution = 5 μL/1 mL of working standard in 1 mL of solvent.
1/100 dilution = 10 μL/1 mL of working standard in 1 mL of solvent
1/50 dilution = 20 μL/1 mL of working standard in 1 mL of solvent

9.2. Set the instrument conditions for analysis to follow guidelines suitable for the instrument. See Appendix A for instrument conditions used successfully.

9.3. Calibrate by sequentially injecting 1 μL of at least five working standards over the appropriate range (e.g., 1/200, 1/100, 1/50, 1/10, STD) with an autosampler.

9.4. Sample preparation of silica gel tubes.

9.4.1. Label two vials with the correct job number and sample number. Designate the back section with (B) and the front section with (F). Transfer the back section from a sample into the appropriately labeled vial. Transfer the front section, including the glass wool, into the other vial.

9.4.2. Add 2.0 mL of desorbing solution to each vial containing media. Cap the vial and allow to stand for two hours.

9.4.3. Using a pipette, transfer equal portions of the sample extract and alkalizing solution to the autosampler vials. Check the pH of the sample using pH paper to ensure that the sample is now basic. If the sample is not basic, use a stronger alkalizing solution.

9.5. Analyze each of the samples, needed spikes, and appropriate laboratory media blanks and continuing calibration standards.

10. CORRECTIVE ACTION

10.1. If interference due to the presence of volatile organic solvents is apparent, use less polar column or change the temperature program.

10.2. If the peak area for any sample is above the linear range of the working standards, dilute with eluent, reanalyze and apply the appropriate dilution factor in the calculations.

10.2.1. The calibration range may also be extended by preparing and analyzing a higher calibration standard followed by a satisfactory continuing calibration standard.

11. CALCULATIONS

11.1. Refer to Appendix A for an example chromatogram and typical retention times.

11.2. The mass(es) of each analyte in each sample are calculated by comparison with a calibration curve (peak vs. µg analyte). Calculations are done by a GC chromatogram computer program or manually by the analyst.

11.3. The mass of each analyte in each sample is calculated based on the following equation:

$$\text{Mass (µg)} = \frac{\text{concentration (µg/mL) x volume correction factor}}{\text{DE correction factor}}$$

where the desorption efficiency factor is based on historical data. No DE correction is necessary if the historical desorption efficiency for an analyte is between 95 and 105%.

11.4. If the sample was diluted, the mass is also multiplied by the dilution factor. For example, for a 1/10 dilution:

$$\text{µg} = \frac{\text{concentration x volume correction factor x 10}}{\text{DE correction factor}}$$

11.5. The results for the client field blank should be subtracted from all sample results. If the client does not submit a blank, the laboratory medial blank is used to correct the client samples.

12. LABORATORY DATA VALIDATION

12.1. See Technikon SOP: XXXXX for data validation procedures. Validation protocols are the same except for the following:

13. REAGENT WASTE DISPOSAL

13.1. Waste is disposed of in accordance with state, county, and federal EPA regulations.

14. METHOD PERFORMANCE

14.1.1. Recoveries of analytes from DE samples should be between 80-110%.

15. REFERENCES

15.1. National Institute of Occupational Safety and Health, NIOSH Manual of Analytical Methods, Fourth Edition, Method 2007 and 2010, Amines, Aliphatic, Issue 2, U.S. Department of Health and Human Services, Cincinnati, OH. August 1994.

Appendix A

Gas Chromatograph instrument conditions successfully used for an HP 5880 or HP 5890

Helium Carrier: 25 mL/min
Hydrogen: 4 mL/min
Air: 80 mL/min

Injection Port: 240°C
Detector: 300°C
Oven: 130°C initial temperature for 5 minutes, ramp at 10°C/min to 240°C, hold at this temperature for XX minutes.

Temperature program may be modified based on analytes requested.

Injection volume: 1 μL

Application Method

DOCUMENT NO.	
MT-7003	
DOCUMENT TITLE: DETERMINATION OF BENZENE AND HYDROCARBONS AS HEXANE IN AIR	PAGE 1 OF 8
DATE ISSUED	SUPERSEDES: NEW
PREPARED BY: C. Hornsby	
APPROVED BY	APPROVED BY
APPROVED BY	

1. SCOPE AND APPLICATION

1.1. This method is applicable for the determination of organic vapors adsorbed from air onto activated charcoal tubes.

1.2. The analytes may be collected on the following types of media:

150 mg coconut charcoal sorbent tubes
600 mg coconut charcoal sorbent tubes
1000 mg charcoal tube
Orbo 32 small to large
Anasorb CMS/747
Orbo 90-91

2. SUMMARY OF METHOD

2.1. The organic vapors are desorbed and analyzed by Gas Chromatography with a Flame Ionization Detector (GC/FID). Quantification is performed by comparison of a known calibration curve of analytes to the samples (peak area or height vs. μg analyte).

3. INTERFERENCES

3.1. Compounds with the same or similar GC retention times can interfere. GC parameters may be changed to circumvent interferences such as altering the type of column, length of column, or operating conditions.

3.2. Consult the specific method reference for optimal GC conditions for the analytes to be analyzed.

3.3. Contamination of the sorbent tube with compounds of interest is commonly encountered. Use care in the preparation, storage, and handling of tubes to minimize this problem.

4. APPARATUS AND MATERIALS

4.1. The following columns and their equivalents are used routinely for these anlayses

4.1.1. DB-1, 5 μm film thickness, 0.32 mm ID, 30 or 60 m length.

4.1.2. DB-5, 5 μm film thickness, 0.32 mm ID, 60 m length.

4.1.3. Stabilwax, 5 μm film thickness, 0.32 mm ID, 60 m length.

4.1.4. Nukol, 5 μm film thickness, 0.32 mm ID, 30 m length.

4.2. Gas Chromatograph with Flame Ionization Detector (GC/FID)

4.3. Personal computer (PC) with chromatography software.

4.4. Microsyringes

4.5. Amber vials with Teflon-lined screw caps

4.6. Autosampler vials with Teflon-lined crimp caps

4.7. Volumetric flasks, class A

4.8. Auto dispenser

4.9. Pipette bulbs and disposable pipettes.

4.10. Preparation of other desorption solvents may require the use of additional glassware/ equipment.

5. REAGENTS

5.1. Carbon Disulfide (low benzene)

5.2. Neat materials, analytical reagent grade purchased as certified materials.

5.3. Helium – purified

5.4. Hydrogen – purified

5.5. Air – compressed, filtered

6. SAMPLE SHIPMENT, PRESERVATION, AND STORAGE.

6.1. Following sample collection, the ends of the tubes are capped with the caps provided. Samples are shipped to the laboratory with ice.

6.2. Samples are stored in the laboratory in a refrigerator designated for volatile samples at 4°C until analysis.

7. HEALTH AND SAFETY

7.1. Refer to the Material Safety Data Sheets (MSDS) for information on toxicity and potential hazards of solvents/chemicals used during this procedure.

7.2. Carbon disulfide is toxic and an acute fire explosion hazard (flash point = -30°C); all work must be done in a hood.

8. QUALITY CONTROL

8.1. The desorption efficiencies determine the measure of accuracy of the analytical procedure by comparing known concentrations against % recovered.

$$\% \text{ recovered} = \frac{\mu g \text{ recovered}}{\mu g \text{ spiked}} \times 100\%$$

Desorption efficiencies are determined for each combination of analytes, solvent, and media types. The initial desorption efficiency study consists of duplicate spikes at three levels corresponding to a low, mid, and high calibration level. Duplicate spikes are prepared at one of the calibration levels with each batch for subsequent analyses.

8.1.1. For 150 mg tubes, remove and discard the back sorbent section and inject 20 µL of the standard (low calibration level), 20 µL of a 10X standard (mid-range calibration), or 2 µL of neat material of a specific analyte (high calibration level) into the front sorbent charcoal using the appropriate syringe. Cap the sample tubes.

8.1.2. For 600 mg tubes, remove and discard the back sorbent section and inject 40 µL of the standard (low calibration level), 40 µL of a 10X standard (mid-range calibration), or 4 µL of neat material of a specific analyte (high calibration level) onto the media using the appropriate syringe.

8.1.3. Store all spiked media in a refrigerator and allow 12 hours for adsorption.

8.1.4. Other levels may be spiked as necessary.

8.2. Prepare one laboratory media blank for each media type.

8.3. A verification standard must also be prepared from the same neat material as used for the working standard. The verification standard and the working standard must be linear within ±10%. See Section 9.2 for preparation instructions.

8.4. A continuing calibration standard (see preparation in Section 9.1.4.1) will be run every 10 injections. The overall response must be within ±10% of each.

9. PROCEDURE

9.1. Stock Standard preparation

9.1.1. The working standard that is prepared is 5 µL neat analyte in 10 mL of solvent (5µL/10 mL).

9.1.2. Fill an appropriate volumetric flask (e.g., 10 mL) to approximately 9 mL with the desired solvent.

9.1.3. Using a syringe, add 5 µL of neat analyte to the volumetric flask.

9.1.3.1. Rinse the syringe at least 4 times with the neat material.

9.1.3.2. After drawing back past the 5 µL mark, position the syringe upside-down making sure there are no trapped air bubbles.

9.1.3.3. Carefully push the plunger to the 5 μL mark and add the material to the volumetric flask.

9.1.3.4. Rinse the syringe with solvent between analytes.

9.1.3.5. Fill the volumetric flask to the mark. Cap and invert several times.

9.1.4. Dilutions are prepared from this working standard.

9.1.4.1. A 1/10 dilution is used for the continuing calibration standard. For example, fill the volumetric flask with approximately 8 mL of desired solvent then pipette 1 mL of the working standard into a 10 mL volumetric flask and dilute to volume with the appropriate solvent.

9.1.4.2. Standard dilutions can be made directly in autosampler vials. Pipette 1 mL of solvent into each vial. Using a syringe, add the appropriate volume of the working standard to each autosampler vial for the chosen dilutions as stated below:

1/200 dilution = 5 μL/1 mL of working standard in 1 mL of solvent.
1/100 dilution = 10 μL/1 mL of working standard in 1 mL of solvent
1/50 dilution = 20 μL/1 mL of working standard in 1 mL of solvent

9.2. When a new working standard is prepared, it must be verified with a standard at 10X the concentration level.

9.2.1. Prepare the 10X standard. For example, add1.0 mL of solvent to an autosampler vial.

9.2.2. Add 5.0 μL of neat material for each analyte to the autosampler vial. Volume correct the 10X standard if more than 4 analytes are to be added (e.g. 5 analytes = 25 μL of neat material; using a syringe, remove 25 μL of solvent from the vial before the analytes are added).

9.2.3. Upon analysis, the 10X standard and the working standard must be linear within ±10%.

9.3. Set gas chromatograph parameters (see Appendix A for suggested conditions).

Note: The temperature program may be modified based on the analyte(s) and the column used.

9.4. Calibrate by sequentially injecting at least five working standards over the appropriate range.

9.5. Transfer each section of charcoal into an appropriately marked vial.

9.6. Add an appropriate volume of desorption solvent, e.i. 1 mL for the 100mg/50mg tubes, cap tightly and allow to stand with occasional shaking for sixty (60) minutes.

9.7. Inject each of the samples, necessary spikes, appropriate laboratory media blanks, and continuing calibration standards into the gas chromatograph.

10. CORRECTIVE ACTION

10.1. If the peak area is above the linear range of the working standards, dilute with eluent, reanalyze and apply the appropriate dilution factor in calculations; or run a higher working standard.

11. CALCULATIONS

11.1. The mass(es) of each analyte in each sample are calculated by comparison with a calibration curve (peak vs. μg analyte). Calculations are done by a GC chromatogram computer program or manually by the analyst.

11.2. The mass of each analyte in each sample is calculated based on the following equation:

$$\text{Mass } (\mu g) = \frac{\text{concentration } (\mu g/mL) \times \text{volume correction factor}}{\text{DE correction factor}}$$

11.3. If the sample is diluted, the mass is also multiplied by the dilution factor. For example, for a 1/10 dilution:

$$\mu g = \frac{\text{concentration x volume correction factor}}{\text{DE correction factor}} \times 10$$

11.4. The results for the client field blank should be subtracted from all sample results. If the client does not submit a blank, the laboratory media blank is used to correct the client samples.

12. REAGENT WASTE DISPOSAL

12.1. Waste is disposed of in accordance with state, county, and federal EPA regulations.

13. REFERENCES

13.1. OSHA Analytical Methods Manual, Organic Methods, "Organic Vapors", Part 1, Vol. 1, Method 7, Revision 1, Organic Methods Evaluation Branch, OSHA Analytical Laboratory, Salt Lake City, UT. 1989.

13.2. National Institute of Occupational Safety and Health, *NIOSH Manual of Analytical Methods*, 4th Ed., Vol. 1, U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 94-113 (1994).

1.1.Appendix A

Gas Chromatograph parameters successfully used:

Helium Carrier: 1-2 mL/min, Splitter Vent: 12-30 mL/min
He FID Makeup Gas: 30 mL/min.
Hydrogen: 30 mL/min
Air: 400 mL/min
Purge Flow: 3-5 mL/min
Linear Velocity: 3.25-3.45 min., for 60 m columns, 1.5-1.7 min., for 30 m columns
Injector, Detector- Oven conditions will vary upon type of column/analytes.