MEETING MINUTES: ROBO SURVEILLANCE PANEL

Meeting: ROBO SP Meeting

Date: July 22, 2021

Location: MS Teams (virtual)

Minutes by: Justin Mills – SP Chair

Actions:

1. Justin Mills to calculate and propose final limits for reference oil 436.
2. Tom Schofield to generate a mock-up and proposal “clean-up” the reference oil table in the LTMS. Only “active” reference oils will remain ROBO section and “obsolete” oils will be moved to Appendix A of the document.
3. Tom Schofield to draft an information letter regarding the acceptance of the alternative ROBO method. To be effective 3 months from today (July 22)
4. Tom Schofield to add the following items to the data dictionary: 1) Nitrogen dioxide option: (L or D) 2) Total NO2 delivered: (numeric, one decimal, 2 digits, units=mL). To be effective 3 months from today (July 22)
5. Justin Mills to tentatively schedule the next ROBO SP meeting for September 23, 2021.

Membership and Attendance:

Ace Glass	Dave Lawrence, Tom Petrocella
Afton	*Shelia Thompson, Jeff Yang, *Todd Dvorak
ASTM TMC	Tom Schofield
BG Products	*Madeleine Dellinger
Chevron Oronite	*Robert Stockwell
ExxonMobil	Dennis Gaal
Infineum	Andy Richie, Sapna Eticala
Intertek	*Joe Franklin, Matt Schlaff, *Rachel Stone
Lubrizol	*Aimee Shinhearl, Jerimiah Westbrook
PetroChina	Li Shaohui , Sun Ruihua, Peng Wang, Xiaogang Li, Xu Li
Evonik Oil Additives	*Justin Mills, *Justin Kontra, *Gabriel Walkup
Vanderbilt Chemicals	*Al Filho, Ron Hiza
SwRI	Becky Grinfield, Joe De La Cruz, *Mike Birke, *Young-Li McFarland
Valvoline	Amol Savant, Kevin Figgatt, Steve Lazzara, *Amy Ross
Koehler Instruments	Raj Shah, Vincent Colantuini
Tannas/Savant	Greg Miller, Ted Selby
General Interest	*Alan Flamberg
Guests	

* Denotes attendance

MEETING MINUTES: ROBO SURVEILLANCE PANEL

Summary:

- Meeting convened at 10:02EDT on July 22, 2021
- No modifications to agenda
- ASTM Antitrust and Recording Policy reviewed
- Membership review and update
    - Madeleine Dellinger of BG Products added
    - Steve Lazzara of Valvoline to retire and to be removed from the SP – Many thanks for his contributions to the SP over the years!
- Meeting minutes from June 24, 2021 SP meeting were accepted (motion made by Joe Franklin and seconded by Alan Flamberg)
- Actions from the June 24th meeting were reviewed.
    - Completed - Justin/Alan to incorporate feedback received on draft revision to ASTM D7528 recirculated among the Surveillance Panel.
    - To be completed by next meeting - Tom Schofield to generate a mock-up and proposal "clean-up" the reference oil table in the LTMS. Only "active" reference oils will remain ROBO section and "obsolete" oils will be moved to Appendix A of the document. (Tom Schofield on vacation. To be revisited next meeting.)
    - Completed - Justin Mills to tentatively schedule the next ROBO SP meeting for July 22, 2021.
- Current status of ROBO
    - Current semester (4/1/2021 through 9/30/21) is running mild (-0.39), but precision in line with target (0.1965).
- TMC reference oils
    - As of July 19, 2021, there are 24 acceptable datapoints for 436. SP agreed with the recommendation to set final limits at our next meeting.
- Dilute Nitrogen Dioxide
    - SP reviewed the latest (and final) draft of the D7528 revision. Alan Flamberg walked the SP through each of the changes that had been made since the prior draft – most of these changes were editorial in nature and the overall "spirit" of the draft remained unchanged.
        - Two numbering errors were identified and corrected during this meeting – in the *Summary of Changes* we corrected the following "addition of Appendix X5 and Appendix A6" to "addition of Appendix X6 and Appendix X7".
    - Following the walkthrough of the changes, Alan Flamberg made a motion **"to accept the (ASTM D7528) method as edited during today's meeting and put it into an information letter. Changes to be effective 3 months from today – tentatively"**
        - The motion was seconded by Joe Franklin.
        - A verbal vote was carried out with all participating parties voting affirmative. There were no negatives or abstains.
        - **The motion carried and has been accepted.**
    - Updating the data dictionary to reflect the addition of dilute NO2 as an alternative was also reviewed. After some discussion, general alignment was reached within the SP. A motion was made by Joe Franklin to **"Update the data dictionary to include the following: 1) Nitrogen dioxide option: (L or D) 2) Total NO2 delivered: (numeric, one decimal, 2 digits, units=mL) → example 2.0. To be effective in 3 months from today to coincide with the effective date of the revised method."**
        - The motion was seconded by Alan Flamberg
        - A verbal vote was carried out with all participating parties voting affirmative. There were no negatives or abstains.
        - **The motion carried and has been accepted.**
- Instatherm reactors
    - At our last SP meeting (June 24), Intertek reported issues with newer flasks "burning out" after ~5 runs. All affected reactors were purchased by Intertek in 2021.
        - Madeleine of BG Products reported a similar issue with one reactor purchased in June 2020. She also reported the reactor was severe and had a lot of deposits before it burned out. ACE Glass replaced it at no cost.
    - Rachel Stone of Intertek is scheduled to have a meeting with ACE Glass in the coming weeks to discuss the root cause. Rachel agreed to share any relevant information at our next SP meeting.

  - Approval of alternative reaction flasks was also briefly discussed; however, it was acknowledged that it may take a lot of effort to demonstrate equivalence.
- Next meeting tentatively scheduled on September 23, 2021. Date may be postponed if necessary.
- Meeting adjourned 11:52EDT

Meeting Outcome:

1. There is enough data to set the final limits for Reference oil 436. Limit setting will be addressed at our next meeting.
2. Dilute NO2 alternative method has been approved by the Surveillance Panel. The effective date will be 3 months from today.
3. The Surveillance Panel accepted a motion to add the following two items to the data dictionary: 1) Nitrogen dioxide option: (L or D) 2) Total NO2 delivered: (numeric, one decimal, 2 digits, units=mL). The effective date will be 3 months from today, coinciding with the effective date of the revised method.
4. Two labs have reported similar “burn out” issues with the Instatherm reaction flask. Ace Glass is investigating.

-End report-

ASTM D7528: Bench Oxidation of Engine Oils by ROBO Apparatus

ROBO Surveillance Panel Meeting

July 22, 2021

Justin Mills

Agenda

- Welcome, ASTM statement
- Review membership of surveillance panel
- Review and approve minutes from previous meetings (see attachment)
- Review and follow-up on actions from June 24th meeting
- Current status of ROBO including statistics
- Dilute nitrogen dioxide
  - Approval of alternative method
  - Data dictionary update
- ~~Reference oil list "clean-up" in LTMS~~ to be discussed at next meeting
- Discussion on Instatherm flasks – one lab has reported issues with newer flasks "burning out" after <5 runs.
- Set next meeting

ASTM Antitrust and Recording Policy

ASTM International is a not-for-profit organization and developer of voluntary consensus standards. ASTMs leadership in international standards development is driven by the contributions of its members: more than 30,000 technical experts and business professionals representing 135 countries.

The purpose of antitrust laws is to preserve economic competition in the marketplace by prohibiting, among other things, unreasonable restraints of trade. In ASTM activities, it is important to recognize that participants often represent competitive interests. Antitrust laws require that all competition be open and unrestricted.

It is ASTMs policy, and the policy of each of its committees and subcommittees, to conduct all business and activity in full compliance with international, federal and state antitrust and competition laws. The ASTM Board of Directors has adopted an antitrust policy which is found in Section 19 of ASTM Regulations Governing Technical Committees. All members need to be aware of and compliant with this policy. The Regulations are accessible on the ASTM website http://www.astm.org/COMMIT/Regs.pdf).

Electronic recording of ASTM meetings is prohibited.

Membership

Ace Glass	Dave Lawrence, Tom Petrocella,
Afton	**Shelia Thompson**, Jeff Yang, Todd Dvorak
BG Products	**Madeleine Dellinger**
ASTM TMC	Tom Schofield
Chevron Oronite	**Robert Stockwell**
ExxonMobil	Dennis Gaal
Infineum	Andy Richie, Sapna Eticala
Intertek	**Joe Franklin**, Matt Schlaff, **Rachel Stone**
Lubrizol	**Aimee Shinhearl**, Jerimiah Westbrook
PetroChina	Li Shaohui , Sun Ruihua, Peng Wang, Xiaogang Li, Xu Li
Evonik Oil Additives	**Justin Mills, Gabe Walkup, Justin Kontra**
Vanderbilt Chemicals	**Al Filho**, Ron Hiza
SwRI	Becky Grinfield, Joe De La Cruz, **Mike Birke**, **Yong-Li McFarland**
Valvoline	Amol Savant, Kevin Figgatt, Steve Lazzara, **Amy Ross**
Koehler Instruments	Raj Shah, Vincent Colantuini
Tannas/Savant	Greg Miller, Ted Selby
General Interest	**Alan Flamberg**
Guests	

Summary of changes:

1. Amy Ross of Valvoline added
2. Jerimiah Westbrook of Lubrizol added
3. Madeleine Dellinger of BG Products added
4. Steve Lazzara of Valvoline to retire – Many thanks for his contributions to the SP over the years!

Motion to accept June 24, 2021 meeting minutes

MEETING MINUTES: ROBO SURVEILLANCE PANEL

Meeting: ROBO SP Meeting

Date: June 24, 2021

Location: MS Teams (virtual)

Minutes by: Justin Mills – SP Chair

Actions:

1. Justin/Alan to incorporate feedback received on draft revision to ASTM D7528 recirculated among the Surveillance Panel.
2. Tom Schofield to generate a mock-up and proposal "clean-up" the reference oil table in the LTMS. Only "active" reference oils will remain ROBO section and "obsolete" oils will be moved to Appendix A of the document.
3. Justin Mills to tentatively schedule the next ROBO SP meeting for July 22, 2021.

Membership and Attendance:

Ace Glass	Dave Lawrence, *Tom Petrocella
Afton	Shelia Thompson, Jeff Yang, *Todd Dvorak
ASTM TMC	*Tom Schofield
Chevron Oronite	Robert Stockwell
ExxonMobil	Dennis Gaal
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Koehler Instruments	Raj Shah, Vincent Colantuini
Tannas/Savant	Greg Miller, Ted Selby
General Interest	*Alan Flamberg
Guests	

* Denotes attendance

MEETING MINUTES: ROBO SURVEILLANCE PANEL

Summary:

- Meeting convened at 10:03EDT on June 24, 2021
- No modifications to agenda
- ASTM Antitrust and Recording Policy reviewed
- Membership review and update
  - Amy Ross (Valvoline) added to SP list.
  - Jerimiah Westbrook (Lubrizol) added to SP list
- Meeting minutes from April 15, 2021 SP meeting were accepted (motion made by Joe Franklin and seconded by Tom Petrocella)
- Actions from the April 15th meeting were reviewed.
  - Reference oil 436 (and corresponding limits) added into LTMS. Use of 438-2 has been suspended.
  - Dilute NO2 workgroup (Alan Flamberg, Matt Schlaff, Justin Mills, Tom Schofield) collected available feedback from SP and incorporated it into latest draft of ROBO method.
- Current status of ROBO
  - ROBO report from June D02.B0.07 meeting was shared. Report indicated that ROBO test was in overall good health with not immediate concerns for the method, parts availability, reference oils, test availability, or severity and precision.
  - Statistics for current semester not available on TMC website. Tom Schofield to investigate (may just require the system to refresh).
    - ***Update*** Issue resolved after our SP meeting. Precision is improving at expense of bias. Stats as posted are:

Period	N-size	Degrees of Freedom	Pooled s	Mean Δ/s
4/1/21 through 9/30/21	55	49	0.1912	-0.41

- TMC reference oils
  - At the April 15th meeting, SP approved limits for 436 as a replacement for 438-2. Since this time, acceptance bands for 436 have been entered into the LTMS document, and usage of 438-2 has been suspended.
    - Final status of 438-2 to be clarified at our next meeting.
- Dilute Nitrogen Dioxide
  - A draft revision of ASTM D7528 (version June 2021) was reviewed with the Surveillance Panel. Feedback on the revisions was collected during the meeting. Many were editorial in nature.
    - Update Footnote #2 to include latest information letter (IL20-1).
    - 6.7.2 "…valve to switch between the two ~~air~~ gas sources"
    - 6.8.2 "Second ~~air~~ gas supply…"
    - 7.1 should be updated to reflect both concentrated and dilute NO2 as reagents.
      - 7.1 Nitrogen Dioxide…7.1.1 Liquid Nitrogen Dioxide…7.1.2 Dilute Nitrogen Dioxide in Air
    - 10.3.1 "Start subsurface ~~pure~~ dry-air flow…"
    - 10.5.3.2 Should include a statement about dilute NO2 equivalent to 2.0 mL +/- 0.1 mL
    - 13.3.6.1.1 Should include a reference to X7
    - X7 Numbering needs to be updated
    - X7 Should also add statement or footnote to that indicates this calculation is not necessary when using a mass flow controller.
  - Once the draft method is accepted by the SP, a TMC information letter along with memo detailing equivalence of dilute NO2 to liquid NO2 will be issued.
    - Drafting this information letter may take longer than usual due to the large amount of changes to include.
  - The data dictionary will also require updating to introduce new items associated with dilute NO2. Updates to the data dictionary may take ~2 months due to need to 30-day beta.
- Updating reference oil table in LTMS
  - It was proposed to update the reference oil table in the LTMS to reflect the status of the reference oil – e.g. active or obsolete. This effort was supported by the SP. Based on current practice in the LTMS, it is likely that "active" oils would remain in ROBO section while "obsolete" oils would be moved to the Appendix A:

MEETING MINUTES: ROBO SURVEILLANCE PANEL

"HISTORY OF LTMS REFERENCE OIL MEANS AND STANDARD DEVIATIONS". One potential issue is that Appendix A does not include acceptance bands and instead just includes the average and standard deviation. SP would prefer to preserve the acceptance bands.
  - Tom Schofield agreed to generate a mock-up and proposal for our next meeting.
- Additional topics
  - Intertek has been experiencing issues with some of the newer Instatherm flasks. Flasks have been burning out after 1-5 runs. No other labs have reported this issue. Ace Glass is investigating.
    - Unfortunately, the SP was not able to discuss this topic any further due to time. This topic will be addressed at the next SP meeting.
- Next meeting tentatively scheduled on July 22, 2021. Date may be postponed if necessary.
- Meeting adjourned 11:39EDT

Meeting Outcome:

1. Reference oil 436 added to LTMS and is actively being assigned. Use of reference oil 438-2 has been suspended.
2. The draft revision to ASTM D7528 was reviewed. Feedback was gathered during the meeting and will be implemented into next draft and recirculated among the SP. If all goes according to plan, we will seek a SP vote at the next meeting.
3. SP agreed that it would be beneficial to "clean-up" the reference oil list for ROBO in the LTMS document. Proposal is to only include "active" reference oils in ROBO section and move "obsolete" oils to Appendix A of the document. Tom Schofield agreed to generate a mock-up and proposal for our next meeting.
4. Next meeting scheduled for July 22, 2021.

-End report-

Actions from June 24th meeting

1) Justin/Alan to incorporate feedback received on draft revision to ASTM D7528 recirculated among the Surveillance Panel.

2) Tom Schofield to generate a mock-up and proposal “clean-up” the reference oil table in the LTMS. Only “active” reference oils will remain ROBO section and “obsolete” oils will be moved to Appendix A of the document.

   – Tom Schofield on vacation. To be revisited next meeting.

3) Justin Mills to tentatively schedule the next ROBO SP meeting for July 22, 2021.

Current status of ROBO

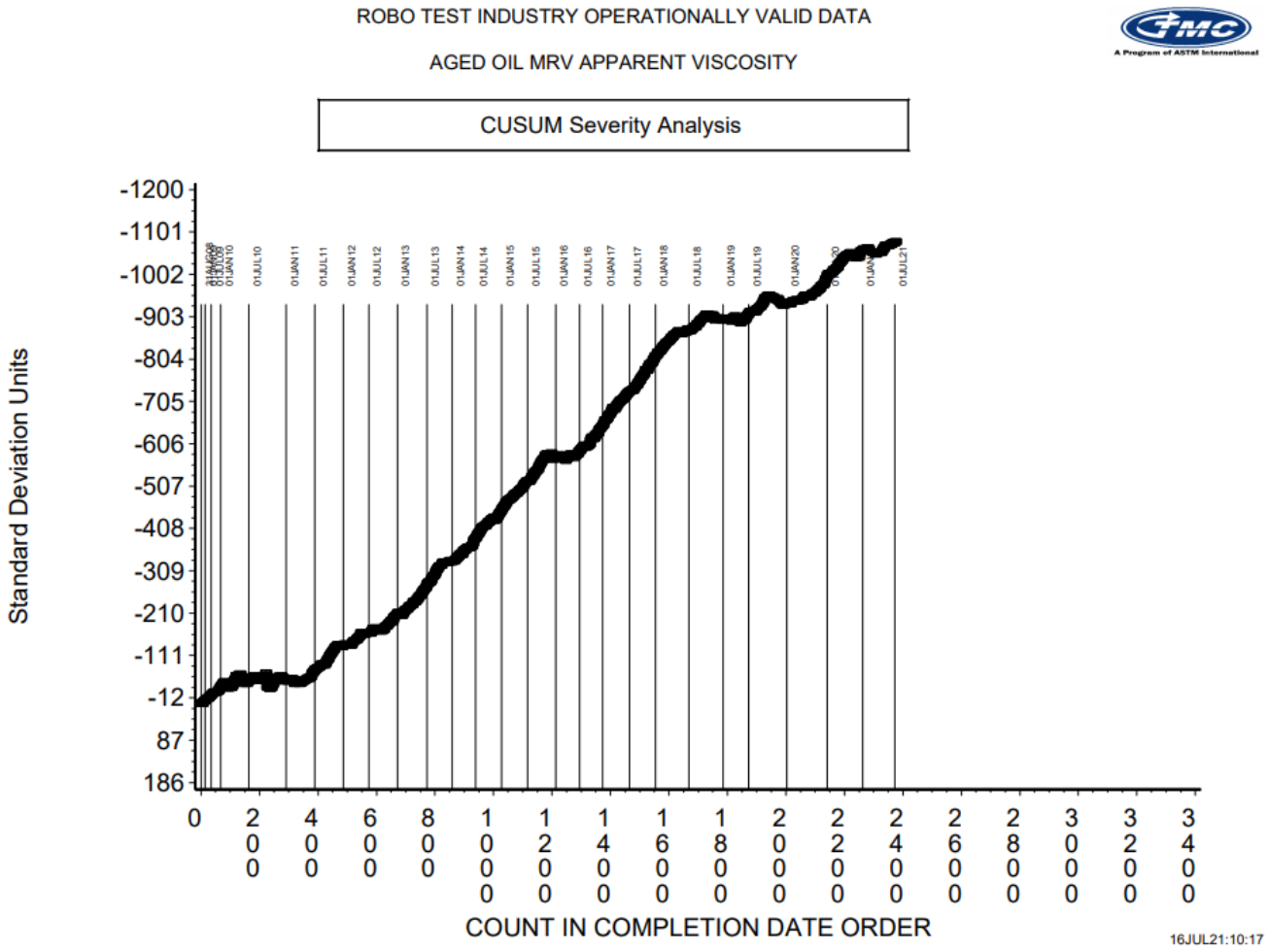
ROBO Industry Statistics

Period	N-size	Degrees of Freedom	Pooled s	Mean Δ/s
Current Targets	49	46	0.1945	------
4/1/17 through 9/30/17	99	95	0.2220	-0.76
10/1/17 through 3/31/18	91	87	0.2367	-0.91
4/1/18 through 9/30/18	126	122	0.2184	-0.49
10/1/18 through 3/31/19	100	96	0.2738	0.04
4/1/19 through 9/30/19	95	91	0.2492	-0.32
10/1/19 through 3/31/20	158	153	0.2723	-0.10
4/1/20 through 9/30/20	119	113	0.2264	-0.76
10/1/20 through 3/31/21	113	108	0.3188	-0.11
4/1/21 through 9/30/21	65	59	0.1965	-0.39

- Precision has improved; however, bias is has shifted mild

Source: http://www.astmtmc.cmu.edu/ftp/refdata/bench/robo/data/statistics.txt (Jul19-2021)

CUSUM severity analysis



Source: http://www.astmtmc.cmu.edu/ftp/refdata/bench/robo/plots/mrv%20INDUSTRY.pdf (July19-2021)

Reference oils

TMC reference oils
Current limits

D7528 (ROBO) Aged Oil MRV Acceptance Bands, mPa·s and ln(mPa·s)

Oil	n	Natural Log Transformed Mean (ln)	Mean in Original Units	s.d. (ln)	95% band in mPa·s Min[1]	95% band in mPa·s Max[1]	95% Bands Min (ln)	95% Bands Max (ln)
434-1	13	10.6599	42,612	0.1672	30,706	59,136	10.3322	10.9876
434-2	36	[2]10.9284	[2]55,737	0.1551	[2]41,126	[2]76,008	[2]10.6244	[2]11.2386
434-3	22	[2]10.8172	[2]49,871	0.1389	[2]37,987	[2]65,473	[2]10.5450	[2]11.0894
435	15	11.4895	97,685	0.2932	[3]60,000	173,546	[3]11.0021	12.0642
435-1	22	11.0416	62,420	0.20295	[4]44570	92910	[4]10.7048	11.4394
436	17	[2]10.3437	[2]31,061	0.1605	22,677	42,544	10.0291	10.6583
438	14	10.2676	28,785	0.2037	19,308	42,912	9.8683	10.6669
438-2	19	[2]10.5404	[2]37813	0.2596	[2]22,734	[2]62,894	[2]10.0316	[2]11.0492

[1] 95% bands in mPa·s are listed for information purposes only, the transformed values will be used to judge acceptance in all cases.
[2] A bias adjustment has been applied to the mean of reference oils 434-2, 434-3, 436 and 438-2 to account for biases observed in the TMC reference data during the periods that each oil target dataset was generated. The 95% confidence range reflects the inclusion of the bias adjustments.
[3] The minimum value for Reference oil 435 is fixed at 60,000 (11.0021 in transformed units) and not a true 95% minimum as calculated from the statistics.
[4] The minimum value for reference oil 435-1 is based on -1.66 standard deviations from the target mean (to match the range previously approved for oil 435 min), so is not actually a 95% confidence range. A 95% confidence range would use 1.96 standard deviations from target mean.

- As of July 19, 2021, there are 24 acceptable datapoints for 436. Recommend we set final limits at our next meeting.

Source: http://www.astmtmc.cmu.edu/ftp/docs/ltms/ltms.pdf 05-2021 version

Dilute NO_2

Dilute nitrogen dioxide
Next steps

July192021 Draft

Path forward to implement dilute NO2 as an alternative to pure NO2 is the following:

1) **Demonstrate equivalence to the SP** → Based on the available data, SP feels confident that dilute NO2 and concentrated NO2 yield comparable results.
2) Develop a procedure for dilute NO2 → 3rd Draft completed.
   - Draft initially shared at April 15th SP meeting. Revisions and subsequent drafts reviewed again June 24 and today.
3) Approve by SP → Seek approval on current version today.
4) Issue information letter allowing use of dilute NO2 as an alternative
   - Information letter will take a while to draft given the number of changes we incorporate
   - Report form changes may take ≥ 3 months.
5) Ballot the recommended changes at ASTM

Motion to accept the (ASTM D7528) method as edited during today's meeting and put it into an information letter. Changes to be effective 3 months from today – tentatively

- Motion made by Alan Flamberg. Seconded by Joe Franklin
- A verbal vote was carried out with all participating parties voting affirmative. There were no negatives or abstains.
- The motion carried and has been accepted.

This slide was added after our meeting to document the motion.

Data dictionary update and report form update

- **the option used to add nitrogen dioxide: liquid or dilute gas (L/D?)**
  - either is OK, but must match previous run if it is a second reference run, otherwise, a new certification is being started
- ~~dilute only: time-averaged subsurface flow rate of the dilute no2 (report to nearest 1 ml/min)~~
  - ~~no set limit, but constrained by other calculations - (explain if not 185 ml/min?)~~
- ~~dilute only: actual time the subsurface air flow was changed from dilute to pure air~~
  - ~~11.0 - 13.0 (10.6.6.1.1 - limit for liquid NO2. Do we want a tighter limit for the dilute? May not be necessary.)~~
- ~~dilute only: concentration of NO2 in air~~
  - ~~1.07 % - 1.19 % by volume in air (6.7.2)~~
- ~~**dilute only:**~~ **total amount NO2 delivered (calculated)**
  - 1.9 - 2.1 mL (10.5.3.2 - liquid equivalent)

Motion to include the following items in the data dictionary with an effective date of 3 months from today

- Nitrogen dioxide option: (L or D)
- Total NO2 delivered: (numeric, one decimal, 2 digits, units=mL) → example 2.0

- Motion made by Joe Franklin. Seconded by Alan Flamberg
- A verbal vote was carried out with all participating parties voting affirmative. There were no negatives or abstains.
- The motion carried and has been accepted.

This slide was added after our meeting to document the motion.

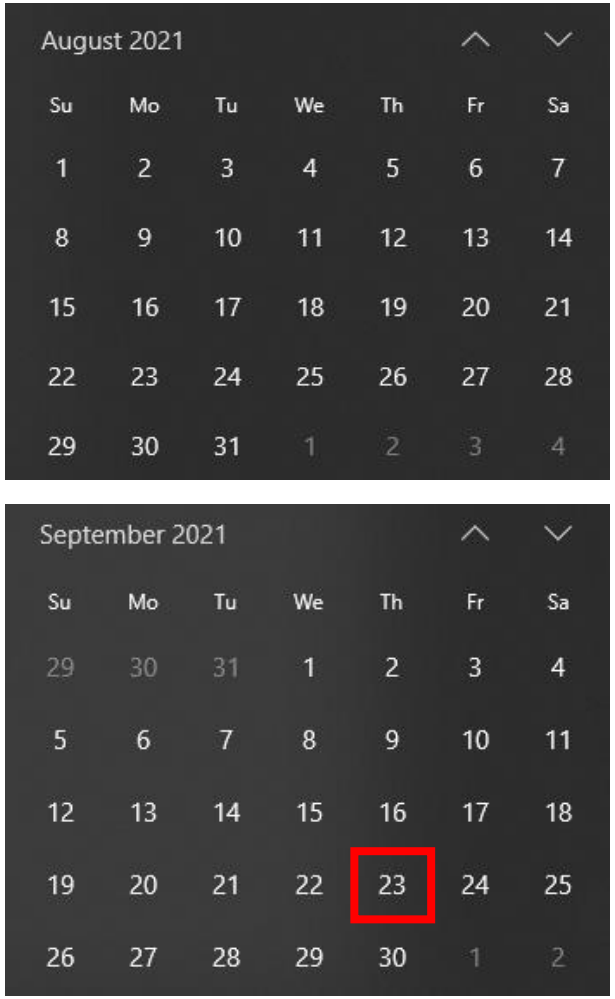
Instatherm update

- One lab has reported issues with newer flasks “burning out” after <5 runs.
- Upon reading the notes from our last meeting, another lab reported similar issues.

Any Additional Topics?

Next Meeting

- Next meeting is tentatively scheduled for September 23rd



Designation: D7528 – 17a

Date: July 19, 2021

To: ROBO Surveillance Panel Subcommittee <AXX.XX> or Main Committee <AXX> members (both for concurrent ballots)

Tech Contact: Alan.Flamberg@gmail.com or Justin.Mills@Evonik.com<Contact Name, email address/phone number>

Work Item #: <Enter Work Item number>

Ballot Action: Revision of D7528

Rationale: Add an alternative way to introduce nitrogen dioxide to the reactor using dilute nitrogen dioxide in air. This option eliminates the need to handle concentrated nitrogen dioxide liquid in the lab.<Enter reasons for proposed ballot action. Include an update on previous ballot history, if applicable>

Standard Test Method for
Bench Oxidation of Engine Oils by ROBO Apparatus[1]

This standard is issued under the fixed designation D7528; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

INTRODUCTION

This test method is written for use by laboratories that make use of ASTM Test Monitoring Center (TMC)[2] services (see Annex A1 – Annex A4).

The TMC provides reference oils, and engineering and statistical services to laboratories that desire to produce test results that are statistically similar to those produced by laboratories previously calibrated by the TMC.

In general, the Test Purchaser decides if a calibrated test stand is to be used. Organizations such as the American Chemistry Council require that a laboratory utilize the TMC services as part of their test registration process. In addition, the American Petroleum Institute and the Gear Lubricant Review Committee of the Lubricant Review Institute (SAE International) require that a laboratory use the TMC services in seeking qualification of oils against their specifications.

The advantage of using the TMC services to calibrate test stands is that the test laboratory (and hence the Test Purchaser) has an assurance that the test stand was operating at the proper level of test severity. It should also be borne in mind that results obtained in a non-calibrated test stand may not be the same as those obtained in a test stand participating in the ASTM TMC services process.

1. Scope *

1.1 This test method describes a bench procedure to simulate the oil aging encountered in Test Method D7320, the Sequence IIIG engine test method. These aged oils are then tested for kinematic viscosity and for low-temperature pumpability properties as described in the Sequence IIIGA engine test, Appendix X1 of Test Method D7320.

1.2 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.2.1 *Exceptions*—There are no SI equivalents for some apparatus in Section 6, and there are some figures where inch units are to be regarded as standard.

1.3 This test method is arranged as follows:

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[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum Products, Liquid Fuels, and Lubricants and is the direct responsibility of Subcommittee D02.B0.07 on Development and Surveillance of Bench Tests Methods.

Current edition approved Oct. 1, 2017. Published October 2017. Originally approved in 2009. Last previous edition approved in 2017 as D7528 – 17. DOI: 10.1520/D7528-17A.

*A Summary of Changes section appears at the end of this standard.

[2] Until the next revision of this test method, the ASTM Test Monitoring Center will update changes in the test method by means of information letters. Information letters may be obtained from the ASTM Test Monitoring Center, 6555 Penn Ave., Pittsburgh, PA 15206-4489. Attention: Administrator. This edition incorporates revisions in all information letters through No. 20-1.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Specific warning statements are given in Sections 7 and 8.

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[3]

D445

D4175

D4485

D4684

[3] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

D5293

D7320

2.2 *SAE Standard:*[4]

SAE J300 Engine Oil Viscosity Classification

3. Terminology

3.1 *Definitions:*

3.1.1 *candidate oil, n*—an oil that is intended to have the performance characteristics necessary to satisfy a specification and is to be tested against that specification. **D4175**

3.1.2 *reference oil, n*—an oil of known performance characteristics, used as a basis for comparison.

3.1.2.1 *Discussion*—Reference oils are used to calibrate testing facilities, to compare the performance of other oils, or to evaluate other materials (such as seals) that interact with oils.

D4175

3.1.3 *non-reference oil, n*—any oil other than a reference oil, such as a research formulation, commercial oil or candidate oil. **D4175**

3.1.4 *test oil, n*—any oil subjected to evaluation in an established procedure. **D4175**

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *aged oil, n*—a test oil after it has been subjected to the 40 h aging process in a ROBO apparatus.

3.3 *Acronyms:*

3.3.1 *ROBO, n*—Romaszewski Oil Bench Oxidation[5]

4. Summary of Test Method

4.1 The test oil is combined with a small amount of iron ferrocene catalyst and placed in a 1 L reaction vessel. That mixture is stirred and heated for 40 h at 170 ºC with air flowing across the liquid surface under negative pressure. In addition, nitrogen dioxide and air are introduced below the reaction surface. After cooling, the oxidized, concentrated test oil is subjected to pertinent viscometric tests. Evaporated oil is condensed in order to weigh it and calculate evaporative loss.

5. Significance and Use

5.1 This bench test method is intended to produce comparable oil aging characteristics to those obtained with ASTM TMC Sequence IIIGA matrix reference oils 434, 435, and 438 after aging in the Sequence IIIG engine test.

5.2 To the extent that the method generates aged oils comparable to those from the Sequence IIIG engine test, the measured increases in kinematic and MRV viscosity indicate the tendency of an oil to thicken because of volatilization and oxidation, as in the Sequence IIIG and IIIGA (see Appendix X1 in Test Method D7320) engine tests, respectively.

5.3 This bench test procedure has potential use in specifications and classifications of engine lubricating oils, such as Specification D4485.

5.4 The results of this test method are valid when seeking qualification of oils against published specifications only when run on a test stand that has successfully met the calibration requirements specified under the TMC's ROBO test monitoring program.

6. Apparatus

6.1 *Balances:*

6.1.1 *Analytical Balance*—Capable of weighing 200 g with a minimum indication resolution of 0.1 g.

6.1.2 *Analytical Balance*—Capable of weighing 0.1 g with a minimum indication resolution of 0.001 g.

6.2 *Fume Hood,* that vents to the outside atmosphere (see Section 8).

[4] Available from SAE International, 400 Commonwealth Drive, Warrendale, PA 15096-0001, http://www.sae.org.

[5] Kinker, B. G., Romaszewski, R. A., and Palmer, P. A., "ROBO–A Bench Procedure to Replace Sequence IIIGA Engine Test," *Journal of ASTM International (JAI),* Vol 4, No. 10, 2007, Paper ID JAI 100916. Available online from www.astm.org.

6.3 *Reaction Vessel* (ACE Glass, Inc. part number D120676),[6,7] a 1 L, thick-walled glass vessel having a nominal 100 mm inner diameter and with a bottom, sample/drain valve. The lower half has an Instatherm[8,7] coating, rated at approximately 400 W, for heating the test mixture. A diagram is shown in Fig. A5.1.

6.4 *Vessel Head*—The vessel head is a stainless steel plate of sufficient diameter to completely cover the lower glass vessel and provide ample material for a sturdy mounting system. Reimel Machine, Inc. part number RMI-1002-DH[9,7] has been shown to be suitable for this application. The vessel head may also be constructed as described in Annex A6. Users may also source some parts from Reimel Machine, Inc. and some in-house. Ensure the plate has a center hole for an agitator shaft and threaded ports to allow filling and for the attachment of air/nitrogen dioxide lines, vacuum control and relief valves, and a temperature probe. Fig. A6.1 defines the locations of these ports. Mill the bottom surface of this stainless steel plate to accept a polytetrafluoroethylene (PTFE) ring seal for centered attachment of the glass vessel as described in Annex A7. Reimel Machine, Inc. part number RMI-1007-DH[9,7] has been found suitable for this purpose.

6.5 *Stirrer Motor*—An electric motor with drill chuck collet capable of sustained operation at 200 r/min ± 5 r/min.

6.6 *Stirrer*—An 8 mm diameter stainless steel rod, 300 mm long with a means of attaching a blade assembly at the bottom. The turbine blade assembly diameter is 2.58 in. (65.5 mm) with 1.4 mm thick blades attached at a 45° pitch with an overall blade height of 0.985 in. (25.0 mm). Construct the stirrer as described in Annex A8. Reimel Machine, Inc. part number RMI-1001-DH[9,7] has been found suitable for this purpose.

6.6.1 Attach the stirrer to the reactor head by means of a packing gland constructed as described in Annex A9. Reimel Machine, Inc. part number RMI-1004-DH[9,7] has been found suitable for this purpose. Attach the stirrer to the stirrer motor by inserting the 8 mm steel rod through the opening in the reactor head and the packing gland, and insert PTFE rope packing to create a seal.

6.6.2 Position the blade 6 mm from the bottom of the vessel.

6.7 *Air Supply System*—A gas source capable of delivering an uninterrupted flow of dry air into the test oil via a subsurface feed throughout the reaction time period. An in-line, desiccant-charged, drying system has been found suitable.

6.7.1 Ensure the subsurface feed tube opening remains below the surface of the test fluid for the duration of the test. Do not place the tube in the drain area of the reaction flask.

NOTE 1—As the amount of test oil remaining at the end of the test is not always known at the beginning of the test, it is advisable to configure the dry-air tube location such that the opening of the tube is as close to the agitator and as close to the bottom of the reactor as practical (without contacting the agitator or blocking the tube opening).

6.7.2 A second gas source consisting of a gas cylinder containing dilute nitrogen dioxide in air may be added along with a valve to switch between the two gas sources. The concentration of nitrogen dioxide in air needed is 1.13 % by volume. See Appendix X6 for how this is derived. The concentration as certified by the supplier must fall in the range of (1.07 to1.19) % by volume.

6.8 Nitrogen dioxide delivery system. There are two options for adding nitrogen dioxide. One uses liquid nitrogen dioxide and the other uses dilute nitrogen dioxide in air.

6.8.1 *Graduated Tube for Liquid Nitrogen Dioxide* (Ace Glass, Inc., part number D120677),[6,7] 12 mL capacity, with 0.1 mL graduations and having appropriate provisions for connection to the reaction vessel's subsurface gas delivery system—see Annex A10 for more details. By receiving liquid phase nitrogen dioxide from a gas bottle, this tube allows measurement of nitrogen dioxide depletion from the tube over the course of the reaction. This graduated tube only used for the liquid nitrogen dioxide option.

6.8.2 *Gas Cylinder Containing Dilute Nitrogen Dioxide in Air.* Second gas source using dilute nitrogen dioxide in dry air as defined in 6.7.2. This is only used for the dilute nitrogen dioxide option.

6.9 *Temperature Control System*—A controller and probe capable of being programmed to control reaction temperature via low output wattage at or below 40 V ac and with an operational hysteresis of 0.1 °C using an on/off algorithm. Alternatively, a proportional-integral-derivative (PID) algorithm may also be used. Position the temperature probe tip so that it is level with the bottom of the turbine blade with a distance of 8 mm between the probe center and the blade edge.

6.9.1 As the temperature may not be uniform throughout the reactor, it is important from the point of view of precision that the temperature is always monitored and controlled at the specified position inside the reactor. When reassembling the reactor for a new run, reposition the probe, if necessary, as it is easily bent.

6.10 *Flow Meters:*

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[6] The sole source of supply of the apparatus known to the committee at this time is Ace Glass, Inc., P.O. Box 688, 1430 NW Blvd., Vineland, NJ 08362-0688.

[7] If you are aware of alternative suppliers, please provide this information to ASTM. Your comments will receive careful consideration at a meeting of the responsible technical committee[1] which you may attend.

[8] Instatherm is a registered trademark of Ace Glass, Inc., P.O. Box 688, 1430 NW Blvd., Vineland, NJ 08362-0688.

[9] The sole source of supply of the apparatus known to the committee at this time is Reimel Machine, Inc., 2575 Wyandotte Rd., Willow Grove, PA 19090.

6.10.1 *Acrylic Block Airflow Meter* (King Instrument Co., 7520 Series, Order number 2C-17),[10] [7] having a scale of 0.4 to 4 Standard Cubic Feet per Minute (SCFM), with 1/4 in. NPT threaded female pipe end. It is used for measuring air flow in 10.3.2. The machined fitting for the top of the flow meter shall accommodate the vacuum line from the condenser to the reactor with a 3/8 in. inside diameter or larger. The machined fitting for the bottom of the flow meter shall accommodate the 1/4 in. vacuum control valve.

NOTE 2—SCFM is the volumetric flow rate of a gas corrected to *standardized* conditions of temperature, pressure, and relative humidity, thus representing a precise mass flow rate. However, the definitions of *standard* conditions vary. In this method, the flow meter is calibrated with air at *standard* conditions defined as a temperature of 70 °F, a pressure of 14.6 psia and 0 % relative humidity.

6.10.2 *Airflow Meter,* with a scale calibrated in mL/min for measuring subsurface airflow of 185 mL/min in 10.3.1 and 10.3.2. Two air flow meters may be used in the dilute nitrogen dioxide configuration depending on the location of the switching valve.

6.10.2.1 A digital mass flow controller may also be used to measure and control the flow rate. This type of flow controller is recommended, but not required, for the dilute nitrogen dioxide in air option.

6.11 *Vacuum System*—A pump with a free air capability of at least 160 L/min is required to ensure a constant air flow across the reaction surface in the vessel of 2.0 SCFM ± 0.1 SCFM with 61 kPa vacuum for 40 h. Instructions for constructing the vacuum plumbing for the vessel are given in Annex A11. As explained in Annex A11, it is critical to follow these instructions precisely.

6.12 *Vacuum Control Valve*—A stainless steel needle valve with 1/4 in. outside diameter tube connections and a flow coefficient (Cv) of 0.37 has been found suitable for this application.

6.13 *Vacuum Trap System*—Supplies coolant at an inlet temperature <20 °C to the vacuum trap condensers in order to remove vapors from the effluent prior to entering (and possibly damaging) the vacuum system and has a means of recovering the distillate for weighing. Redundant (serial) condensers are beneficial as long as the required airflow across the reaction surface is maintained. Annex A12 provides information on two systems that have been found to be satisfactory.

6.14 *Time Controller*—A timing device accurate to 1 min is used to deactivate the heat source.

6.15 *Precision Needle Valve,* having a low Cv for precise control of the flow of nitrogen dioxide. Examples of valves that have been found satisfactory are given in Appendix X3. This valve is used with the liquid nitrogen dioxide option in 6.8.1. It is not required for the dilute nitrogen dioxide option described in 6.8.2.

6.16 *Beaker*—300 mL capacity.

6.17 *Glass Jar*—250 mL capacity which can be sealed.

6.18 *Shaker*—Use either a reciprocal or an elliptical shaker.

6.19 *Assembled ROBO Apparatus*—Fig. X4.1 shows an example of an assembled ROBO apparatus. However, because it is assembled from different components, some of which are site specific (for example, geometry of fume hood, local safety considerations, use of different parts such as temperature controllers, and so forth), there is no standard ROBO apparatus assembly. As an aid to building and setting up a new ROBO apparatus, a package of information is available on the TMC website.[2] This (non-mandatory) information supplements that given in Section 6. An index to the contents of this information package is given in Appendix X5.

7. Reagents and Materials

7.1 *Nitrogen Dioxide*

7.1.1 *Liquid Nitrogen Dioxide —Used with the option in 6.8.1.* Produces a reddish-brown gas with a pungent odor. (**Warning—**VERY TOXIC if inhaled or ingested. Explosive if mixed with combustible material. Irritating to eyes and respiratory system. Danger of very serious irreversible health effects.).

7.1.2 *Dilute Nitrogen Dioxide in Air —Used with the option in 6.7.2.* (**Warning** - Compared to liquid nitrogen dioxide, the exposure risk is greatly reduced, but not negligible.)

7.2 *Iron Ferrocene*—98 % or higher purity. (**Warning—**Do not breathe dust. Harmful if swallowed.)

7.3 *Oil*—100 Neutral, API Group II, for mixing with iron ferrocene catalyst.

7.4 *Cleaning Solvent*—Commercial heptanes, or similar solvents that evaporate without leaving a residue, are suitable. (**Warning—**flammable.)

7.5 *Acetone*—A technical grade of acetone is suitable provided it does not leave a residue upon evaporation. This is used for a final cleaning rinse. Acetone will degrade fluoroelastomer seals and can dissolve or deteriorate acrylics. (**Warning—** flammable.)

7.6 *Dry Air*—Desiccated air is suitable.

[10] The sole source of supply of the apparatus known to the committee at this time is King Instrument Co., 12700 Pala Drive Garden Grove, CA 92841.

7.7 *Reference Oils*—The current TMC reference oils are required for setting up the ROBO apparatus test stand (see Section 9). The TMC[2] maintains and distributes these oils. These oils are formulated or selected to represent specific chemical types or performance levels, or both. See A2.4 for additional information regarding reference oils.

7.7.1 The TMC is responsible for managing a system that ensures the performance and formulation consistency of the reference oils. Store the reference oils in locations where the ambient temperature does not exceed 32 °C. Under these conditions, the expected shelf life of a reference oil is five years. In some circumstances, however, the TMC may specify a shelf life longer than five years. In such cases, the TMC uses documented analysis procedures to justify the longer shelf life.

7.7.2 Unless specifically authorized by the TMC, do not analyze TMC reference oils, either physically or chemically. The testing laboratory tacitly agrees to use the TMC reference oils exclusively in accordance with the TMC's published Policies for Use and Analysis of ASTM Reference Oils, and to run and report the reference oil test according to TMC guidelines.

NOTE 3—Policies for the Use and Analysis of ASTM Reference Oils are available from the TMC.[2]

8. Hazards

8.1 *Specific Hazards*—Due to nitrogen dioxide toxicity, with the exception of weighing, perform steps 10.3 – 10.8 of the procedure in the fume hood. See also 7.1.

9. New and Existing Test Stand Calibration

9.1 Set up qualifying test runs for a new ROBO apparatus test stand. For existing ROBO apparatus test stands, proceed to 9.2.

9.1.1 Obtain the required, current reference oils from the TMC for the purpose of setting up a new ROBO apparatus stand.[2] (See 7.7.2 and Annex A2 for conditions of use for the TMC reference oils.)

9.1.2 Test the assigned reference oils according to the procedure described in Section 10.

9.1.2.1 It is imperative that the vacuum control valve (VCV) set position be set on the first set-up test and not changed again for subsequent set-up qualifying runs.

9.1.2.2 If the VCV set position is changed by more than ±0.125 revolutions after the start of the first qualifying set-up test run, all previous tests in the set-up test sequence are void; repeat the test stand setup runs from 9.1.1 – 9.1.4.

9.1.3 Determine the viscometric properties of the aged reference oils as described in Section 12 and report according to Section 13.

9.1.4 Report test results to the TMC using the standardized reporting protocols (see 9.2.2 and Section 13). Be sure to include all required operational parameters as defined in the reporting protocol data dictionary.

9.1.5 Review all initial set-up results on new instruments and receive approval from the TMC.

9.1.5.1 Test results will be posted to the TMC website. Lab identification will be coded by the TMC for confidentiality of the testing laboratory.

9.1.6 If all the required test stand set-up runs meet the current, approved ROBO TMC calibration requirements[1111] (both operationally and statistically), the TMC will notify the laboratory that it can proceed with calibrating the test stand per 9.2.

9.1.7 If the TMC's review determines that the required test stand set-up runs do not collectively meet the approved requirements (both operationally and statistically), the TMC will notify the laboratory that additional adjustments need to be made to the test stand and one or more of the set-up runs will have to be repeated.

9.2 *Existing Test Stand Calibration:*

9.2.1 *Reference Oil Test Frequency*—The TMC requires test stands to pass periodic calibration verification with reference oils supplied by the TMC. These calibration verification runs are typically run on blind-coded reference oil samples.

9.2.1.1 Prior to conducting a TMC reference oil test for the purpose of stand calibration, procure a supply of reference oil directly from the TMC. (See 7.7.2 and Annex A2 for conditions of use for the TMC reference oils.) The reference oils are usually supplied directly to a testing laboratory with blind-coded identification numbers to ensure that the laboratory is not influenced by prior knowledge of a reference oil's acceptable performance results in assessing the test results. The TMC will determine which specific reference oil or oils the laboratory shall test in accordance with the calibration requirements.

9.2.1.2 Initial calibration verification of a new test stand or repeated consecutive unacceptable calibration verifications on a test stand requires passing two consecutive TMC reference oil tests.

9.2.1.3 Certain operational changes to the test stand, as specified in the TMC calibration requirements,[11] voids the TMC test stand calibration status and requires passing two consecutive TMC reference oil tests to re-verify the calibration status of the modified test stand.

[11] The ROBO TMC Calibration Requirements document is available at: http://www.astmtmc.cmu.edu/ftp/docs/bench/robo/procedure_and_ils/20170713_ROBO_TMC_Calibration_Requirements.pdf

9.2.1.3.1 The same nitrogen dioxide delivery configuration must be used to re-verify the calibration status and then continued to be used for subsequent certified runs.

9.2.1.4 During the time of conducting a reference oil test on one test stand, non-reference oil tests may be conducted on other previously calibrated stands.

9.2.2 *Test Numbering:*

9.2.2.1 The test number shall follow the format *AAA-BB-CCCC*. *AAA* represents the test stand identification. *BB* represents the number of tests since last reference. *CCCC* represents the total number of tests on the stand. As an example, 6-10-175 represents the 175 test on Stand 6 and the tenth test since the last reference. Consecutively number all tests on a given stand.

9.2.3 *Reporting of Reference Oil Test Results*—Report the results of all reference oil tests to the TMC according to the following instructions:

9.2.3.1 Transmit results according to the ROBO Standardized Report Forms and Data Dictionary[12] to the TMC within five days of test completion via electronic data transfer protocol as outlined in the Data Communication Committee, Electronic Test Report Transmission Model (ETRTM).[13]

NOTE 4—Be sure to collect data on all the required parameters defined in the ROBO Standardized Data Dictionary[12] (see Section 13). Validity evaluation of test results cannot be made if critical evaluation parameters are missing.

9.2.4 *Evaluation of Reference Oil Test Results*—The TMC evaluates the reference oil test results for both operational validity and statistical acceptability. The TMC may consult with the test laboratory in case of difficulty, as follows:

9.2.4.1 Upon receipt of the reference oil test results from the test laboratory, the TMC evaluates the laboratory's reported operational parameters for compliance with the current test method. For operationally valid tests, the TMC then evaluates the pass/fail parameters for statistical validity. The TMC sends a test confirmation report to the test laboratory indicating the overall validity of the calibration test results, and disclosing the non-blind industry reference oil code.

9.2.4.2 In the event the reference oil test is unacceptable, the test laboratory shall provide an explanation of the problem relating to the failure. If the problem is not obvious, carry out operational re-checks (instrumentations, settings, and procedures). Following the re-checks, the TMC assigns another reference oil for testing by the laboratory. If this reference oil test is unacceptable, a reassessment of the stand setup as described in 9.1 may be necessary.

9.2.4.3 It is recognized that a certain percentage of calibration tests will fall outside the acceptance limits because of the application of statistics in the development of the acceptance limits. The TMC decides, with consultation as needed with industry experts (testing laboratories, members of the ASTM Technical Guidance Committee, the surveillance panel, and so forth), whether the reason for any failure of a reference oil test is a false alarm, testing apparatus, testing laboratory, or industry-related problem. The ROBO surveillance panel adjudicates all industry problems.

9.2.5 *Reference Oil Accountability:*

9.2.5.1 Laboratories conducting calibration tests are required to provide a full accounting of the identification and quantities of all reference oils used.

9.2.5.2 With the exception of analysis required in this test method, no additional physical or chemical analysis of new or used reference oils is permitted without the express permission of the TMC. (See 7.7.2 and Annex A2 for conditions of use for the TMC reference oils.)

10. Procedure

10.1 *Vacuum Control Valve Setting*—For a new ROBO apparatus test stand, set the vacuum control valve as described in Annex A13. The control valve setting is critical as it affects the severity of the test. For all subsequent runs involving test oils, use exactly the same control valve setting to that used during the last successful TMC calibration verification run.

10.2 *Catalyst Preparation:*

10.2.1 Weigh 0.1 g ± 0.001 g of iron ferrocene (see warning in 7.2) into an appropriate container such as a 250 mL glass jar.

10.2.2 Add 99.9 g ± 0.1 g of API Group II 100 Neutral oil to obtain 0.100 % ± 0.001 % (mass) iron ferrocene.

10.2.3 Mix thoroughly, until the catalyst is completely in solution as determined by a lack of visible particles.

NOTE 5—This may take 1 h or more.

10.3 *Vessel Seal Check:*

10.3.1 Start subsurface dry-air flow at a rate of 185 mL/min.

10.3.2 On an assembled vessel, install the acrylic block flow meter between the top connection of the vacuum control valve and the vacuum source. Apply vacuum to the vessel and block the vacuum relief orifice long enough to assure the system will attain 85 kPa with a subsurface airflow of 185 mL/min.

[12] The ROBO Standardized Report Forms and Data Dictionary specification is available at: ftp://ftp.astmtmc.cmu.edu/datadict/robo/current/.

[13] The Data Communication Committee, Electronic Test Report Transmission Model (ETRTM) document is available at: ftp://ftp.astmtmc.cmu.edu/docs/datacommunicationscommittee/electronic_test_report_transmission_specification/.

10.3.2.1 The acrylic block air flow meter shall read less than 0.6 SCFM.

10.4 *Preset Vacuum Flow*—With the vacuum still applied to the vessel, set the air flow through the reactor to 2.0 SCFM ± 0.1 SCFM by bleeding air, if needed, into the vacuum line between the vacuum source and the condenser. Maintain the vacuum pressure at 61 kPa ± 1.7 kPa by adjusting the vacuum relief valve. Once these parameters are set, shut off the vacuum and remove the acrylic block flow meter from the system.

10.5 *Sample Preparation and Charging Nitrogen Dioxide:*

NOTE 6—Steps 10.5.1 – 10.5.3 may be carried out in any order or simultaneously.

10.5.1 *Sample Preparation*—Introduce 3.0 g ± 0.1 g of prepared iron ferrocene catalyst solution and 197.0 g ± 1.0 g test oil to the reaction vessel. See Appendix X1 for suggested mixing procedures. If the direct weighing procedure (X1.1.2) is used, do the vessel seal check (10.3) and the preset vacuum flow (10.4) procedure after the apparatus is reassembled.

NOTE 7—The total mass of oil in the reactor is 200 g ± 1.0 g (197.0 g ± 1.0 g from the test oil and 3.0 g from the catalyst solution).

10.5.1.1 Start the stirrer motor and agitate at 200 r/min ± 5 r/min.

10.5.2 Make the electrical connections to the heater. (**Warning—**To avoid electric shock and possible ignition spark, check that the power is de-energized before making electrical connections.)

10.5.3 *Charging Nitrogen Dioxide*

10.5.3.1 *Liquid nitrogen dioxide option only.* Transfer 2.0 mL ± 0.1 mL of liquid nitrogen dioxide (see Section 8 and warning in 7.1) into the graduated tube. See Appendix X2 for examples of how the transfer may be made.

10.5.3.2 *Dilute nitrogen dioxide option only.* The amount of nitrogen dioxide introduced can be calculated. An equivalent to 2.0 mL ± 0.1 mL of liquid nitrogen dioxide is required. See Appendix X.6 for example calculation.

10.6 *Oil Aging:*

10.6.1 *General*—Begin the oil aging by setting the time and temperature and turning on the vacuum.

10.6.1.1 Complete steps 10.6.2 – 10.6.5 within 1 min; the order in which they are carried out is not important.

10.6.2 Set the time controller to 40 h to initiate the oil aging.

10.6.3 Set the temperature controller to 170 °C and commence heating.

10.6.4 Adjust the temperature controller voltage output to 25 V to 40 V.

10.6.5 Turn the vacuum system on.

10.6.6 Start the nitrogen dioxide flow.

10.6.6.1 For the liquid nitrogen dioxide option, immediately after the previous steps, adjust the nitrogen dioxide precision needle valve to allow introduction of nitrogen dioxide in a controlled and gradual manner into the inlet flow stream. Ensure that the nitrogen dioxide is completely depleted from the tube and introduced into the reactor within 12 h ± 1 h.

10.6.6.1.1 Because changes to the nitrogen dioxide flow rate can affect precision, it is imperative that nitrogen dioxide be introduced to the reactor in a controlled and gradual manner. Using a flow rate target of 0.167 mL/h, monitor nitrogen dioxide depletion closely in the first 2 h to 4 h, the aim being to introduce 0.5 mL during that time period. Introduce the remaining 1.5 mL at a similar flow rate, ensuring that the total of 2.0 mL is delivered between 11 h and 13 h. A run is invalid if the flow of nitrogen dioxide exceeds 0.5 mL during any 1 h period.

10.6.6.2 For the dilute nitrogen dioxide option, switch to dilute nitrogen dioxide for 12.0 hours. A run is invalid if the flow of dilute nitrogen dioxide in air deviates from the required 185 ml/min by more than 6 % during at any of the observations. At least 6 observations during the first 6 hours of the air flow must be made and recorded with the last observation being made at about 6 hours. The air flow may be adjusted at these times. If all of the readings before adjustments are within 5 % of 185 ml/min, then no more observations are required. If the air flow deviates by more than 4% during the first 6 hours, then six more observations are required from hours 6-12. After 12.0 hours, switch back to the dry-air supply for the remaining of the test.

10.6.6.2.1 If any deviations from 185 ml/min of more than 2 ml/min were observed (or calculated at the 12 hr switching time), then calculate and report the time-averaged flow rate. See Appendix X7 for examples.

10.7 *Shutdown:*

10.7.1 At the end of the 40 h cycle, allow the system to cool to room temperature while maintaining the airflow and agitation.

10.7.2 Turn off the vacuum. (The vacuum flow can be turned off at any time after completion of the 40 h cycle.) Bleed the pressure by opening a port, for example, the sample addition port. Drain the aged oil into a suitable container.

10.8 *Mass Percent Volatiles Collected:*

10.8.1 Drain the condensed liquid from the vacuum trap system into a tared vessel. Determine and record the mass of the condensed liquid to the nearest 0.1 g.

10.8.2 Calculate as follows:

$$\text{Mass \% volatiles, \% m/m} = 100\,\frac{\text{M(volatiles)}}{\text{M(fresh)}} \qquad (1)$$

where:

M(fresh) = 200 g = the mass of fresh oil added to the reactor in 10.5.1, and

M(volatiles) = mass, g, of condensate collected in 10.8.1.

NOTE 8—The significance of the % volatiles parameter is under investigation.

11. Cleaning

11.1 Clean the reaction vessel with cleaning solvent (see warning in 7.4).

11.1.1 Scrub any residual material off the glass surface while taking care not to scratch the inside of the vessel. Perform a final rinse with acetone (see warning in 7.5).

11.2 Clean the vacuum control valve.

11.2.1 Flush the valve with cleaning solvent or carburetor cleaner, followed with an acetone rinse to remove and avoid any carbon deposits that could reduce or plug the valve orifice.

11.2.2 Additional optional cleaning may be needed in cases where there is insufficient vacuum flow (see 10.4). If vacuum flow is sufficient, skip to step 11.3.

11.2.2.1 Disassemble the valve and remove any carbon deposits from the plug and inside seat of the valve body.

11.2.2.2 Flush as in 11.2.1.

11.2.2.3 Reassemble the vacuum control valve, ensuring that the valve setting is at exactly the same position to that used during the last successful TMC calibration verification run.

11.3 Clean the underside of the reactor cap and all shafts or probes protruding downward into the vessel with cleaning solvent and a lightweight, lint-free towel. Rinse with acetone.

11.4 Ensure that subsurface air supply lines are clear, then clean them with cleaning solvent and reassemble when dry.

11.5 Clean the acrylic block flow meter with cleaning solvent. Do not use acetone which can dissolve or deteriorate acrylics.

12. Calculations and Determination of Test Results

12.1 *Increase in Kinematic Viscosity at 40 °C:*

12.1.1 Calculate as follows:

$$\text{Percent viscosity increase (PVIS)} = 100\,\frac{[\text{KV(aged)} - \text{KV(fresh)}]}{\text{KV(fresh)}} \quad (2)$$

where:

KV(aged) = kinematic viscosity, mm^2/s, at 40 °C of the aged oil as determined by Test Method D445, and

KV(fresh) = kinematic viscosity, mm^2/s, at 40 °C of the fresh oil as determined by Test Method D445.

12.2 *Low-Temperature Viscometric Properties:*

12.2.1 Using Test Method D5293, measure the Cold Cranking Simulator (CCS) viscosity of the ROBO-aged oil at the temperature specified for the SAE W grade of the fresh oil. This temperature can be found in the SAE J300 Viscosity Classification System (hereafter referred to as SAE J300).

12.2.1.1 If the measured CCS viscosity is less than or equal to the maximum CCS viscosity specified in SAE J300 for the SAE W grade of the fresh oil, measure the MRV viscosity by Test Method D4684 at the MRV temperature specified in SAE J300 for the SAE W grade of the fresh oil.

12.2.1.2 If the measured CCS viscosity is higher than the maximum CCS viscosity specified in SAE J300 for the SAE W viscosity grade of the fresh oil, measure the MRV viscosity by Test Method D4684 at 5 °C higher than the MRV temperature specified in SAE J300 for the original SAE W viscosity grade of the fresh oil (that is, at the MRV temperature specified in SAE J300 for the next higher SAE W viscosity grade).

13. Report

13.1 *Report Forms*—For TMC reference oil tests, use the standardized report form set and data dictionary.

NOTE 9—Report the non-reference oil test results on these same forms if the results are intended to be submitted as candidate oil results against a specification.

13.1.1 Report reference oil test results to the TMC according to the ETRTM protocols described in 9.2.3.1.

13.2 *Reporting Units*—Report results in SI units.

13.3 Report the following:

13.3.1 Kinematic viscosity at 40 °C, by Test Method D445, of the test oil before and after aging.

13.3.1.1 Report to two decimal places for viscosities between 10 mm^2/s and 100 mm^2/s and to one decimal place for viscosities >100 mm^2/s.

13.3.2 Percent increase in kinematic viscosity at 40 °C after aging (PVIS)—see 12.1.

13.3.2.1 Report to nearest 0.1 %.

13.3.3 SAE W grade of the fresh oil.

13.3.4 The CCS viscosity and temperature of measurement of the ROBO-aged oil by Test Method D5293.

13.3.5 The MRV viscosity, yield stress and temperature of measurement of the aged oil by Test Method D4684—see 12.2.1.1 and 12.2.1.2.

13.3.6. The option used to add nitrogen dioxide. Liquid nitrogen dioxide or dilute nitrogen dioxide.

13.3.6.1 If the dilute nitrogen dioxide option was used, also report:

13.3.6.1.1 The time-averaged subsurface dilute nitrogen dioxide in air flow rate to the nearest 1 ml/min.

13.3.6.1.2 Report the actual time the subsurface air was changed from dilute nitrogen dioxide in air to dry-air. Report to the nearest 0.1 hour.

13.3.6.1.4 Report the concentration of nitrogen dioxide in air as certified by the supplier. If more than one nitrogen dioxide cylinder was used during the ROBO test, report a time-averaged concentration.

13.3.6.1.5 Calculate and report the total amount of nitrogen dioxide delivered to the reactor. See Appendix X.7 for an example calculation.

14. Precision and Bias[14]

14.1 *Precision*—The precision of this test method as determined by the statistical examination of the interlaboratory tests results is given in Table 1.

TABLE 1 Test Precision[A]

Variable	Intermediate Precision		Reproducibility	
	$S_{i.p.}$[B]	i.p.[C]	S_R[B]	R[C]
PVIS[D]	0.191	0.535	0.267	0.748
MRV viscosity[D]	0.25	0.70	0.40	1.12

[A] These statistics are based on results obtained from an interlaboratory program in which seven samples were tested in seven laboratories on ten test rigs (see 14). The samples consisted of SAE 5W-XX and 10W-30 multigrade engine oils including ASTM Test Monitoring Center Reference Oils 434, 435, and 438.

[B] S = Standard deviation.

[C] This value is obtained by multiplying the standard deviation by 2.8.

[D] The original units for PVIS are percent viscosity increase. The original units for MRV viscosity are mPa·s. These parameters are transformed using ln(result). When comparing two test results on these parameters, first apply this transformation to each test result. Compare the absolute difference between the transformed results with the appropriate (intermediate precision or reproducibility) precision limit.

14.1.1 *Intermediate Precision Conditions*—Conditions where test results are obtained with the same test method using the same oil, with changing conditions such as operators, measuring equipment, test apparatus, and time.

NOTE 10—Intermediate precision is the appropriate term for this test method, rather than repeatability, which defines more rigorous within-laboratory conditions.

14.1.1.1 *Intermediate Precision Limit (*i.p.*)*—The difference between two results obtained under intermediate precision conditions that would in the long run, in the normal and correct conduct of the test method, exceed the values shown in Table 1 in only one case in twenty. When only a single test result is available, the Intermediate Precision Limit can be used to calculate a range (test result ± Intermediate Precision Limit) outside of which a second test result would be expected to fall about one time in twenty.

14.1.2 *Reproducibility Conditions*—Conditions where test results are obtained with the same test method using the same test

[14] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D02-1660. Contact ASTM Customer Service at service@astm.org.

oil in different laboratories with different operators using different equipment.

14.1.2.1 *Reproducibility Limit (*R*)*—The difference between two results obtained under reproducibility conditions that would, in the long run, in the normal and correct conduct of the test method, exceed the values in Table 1 in only one case in twenty.

14.2 *Bias*—No estimate of the bias for this procedure is possible because the performance results for an oil are determined only under the specific conditions of the test and no absolute standards exist.

14.3 *Dilute nitrogen dioxide option effect on precision and bias* — The precision and bias in sections 14.1 – 14.2 were determined with the original liquid nitrogen dioxide option. The ASTM ROBO Surveillance Panel approved the use of the dilute nitrogen dioxide option based on the limited data obtained from which no effect on precision or bias was detected.[2]

15. Keywords

15.1 evaporative loss; low-temperature pumpability; MRV viscosity; oil aging; oil oxidation; oil viscosity; ROBO test; sequence IIIG test; sequence IIIGA test; volatiles

ANNEXES

(Mandatory Information)

A1. ASTM TEST MONITORING CENTER: ORGANIZATION

A1.1 *Nature and Functions of the ASTM Test Monitoring Center (TMC)*—The TMC is a non-profit organization located in Pittsburgh, Pennsylvania and is staffed to: administer engineering studies; conduct laboratory inspections; perform statistical analyses of reference oil test data; blend, store, and ship reference oils; and provide the associated administrative functions to maintain the referencing calibration program for various lubricant tests as directed by ASTM Subcommittee D02.B0 and the TMC Executive Committee. The TMC coordinates its activities with the test sponsors, the test developers, the surveillance panels, and the testing laboratories. Contact TMC through the TMC Director at:

ASTM Test Monitoring Center
6555 Penn Avenue
Pittsburgh, PA 15206-4489
www.astmtmc.cmu.edu

A1.2 *Rules of Operation of the ASTM TMC*—The TMC operates in accordance with the ASTM Charter, the ASTM Bylaws, the Regulations Governing ASTM Technical Committees, the Bylaws Governing ASTM Committee D02, and the Rules and Regulations Governing the ASTM Test Monitoring System.

A1.3 *Management of the ASTM TMC*—The management of the Test Monitoring System is vested in the Executive Committee elected by Subcommittee D02.B0. The Executive Committee selects the TMC Director who is responsible for directing the activities of the TMC.

A1.4 *Operating Income of the ASTM TMC*—The TMC operating income is obtained from fees levied on the reference oils supplied and on the calibration tests conducted. Fee schedules are established by the Executive Committee and reviewed by Subcommittee D02.B0.

A2. ASTM TEST MONITORING CENTER: CALIBRATION PROCEDURES

A2.1 *Reference Oils*—These oils are formulated or selected to represent specific chemical, or performance levels, or both. They are usually supplied directly to a testing laboratory under code numbers to ensure that the laboratory is not influenced by prior knowledge of acceptable results in assessing test results. The TMC determines the specific reference oil the laboratory shall test.

A2.1.1 *Reference Oil Data Reporting*—Test laboratories that receive reference oils for stand calibration shall submit data to the TMC on every sample of reference oil they receive. If a shipment contains any missing or damaged samples, the laboratory shall notify the TMC immediately.

A2.2 **Calibration Testing**

A2.2.1 Full-scale calibration testing shall be conducted at regular intervals. These full scale tests are conducted using coded reference oils supplied by the TMC. It is a laboratory's responsibility to keep the on-site reference oil inventory at or above the minimum level specified by the TMC test engineers.

A2.2.2 *Test Stands Used for Non-Standard Tests*—If a non-standard test is conducted on a previously calibrated test stand, the laboratory shall conduct a reference oil test on that stand to demonstrate that it continues to be calibrated, prior to running

standard tests.

A2.3 *Reference Oil Storage*—Store reference oils under cover in locations where the ambient temperature is between –10 °C and +50 °C.

A2.4 *Analysis of Reference Oil*—Unless specifically authorized by the TMC, do not analyze TMC reference oils, either physically or chemically. Do not resell ASTM reference oils or supply them to other laboratories without the approval of the TMC. The reference oils are supplied only for the intended purpose of obtaining calibration under the ASTM Test Monitoring System. Any unauthorized use is strictly forbidden. The testing laboratory tacitly agrees to use the TMC reference oils exclusively in accordance with the TMC's published Policies for Use and Analysis of ASTM Reference Oils, and to run and report the reference oil test results according to TMC guidelines. Additional policies for the use and analysis of ASTM Reference Oils are available from the TMC.

A2.5 *Conducting a Reference Oil Test*—When laboratory personnel are ready to run a reference calibration test, they shall request an oil code via the TMC website.

A2.6 *Reporting Reference Oil Test Results*—Upon completion of the reference oil test, the test laboratory transmits the data electronically to the TMC, as described in Section 13. The TMC reviews the data and contacts the laboratory engineer to report the laboratory's calibration status. All reference oil test results, whether aborted, invalidated, or successfully completed, shall be reported to the TMC.

A2.6.1 All deviations from the specified test method shall be reported.

A3. ASTM TEST MONITORING CENTER: MAINTENANCE ACTIVITIES

A3.1 *Special Reference Oil Tests*—To ensure continuous severity and precision monitoring, calibration tests are conducted periodically throughout the year. Occasionally, the majority or even all of the industry's test stands will conduct calibration tests at roughly the same time. This could result in an unacceptably large time frame when very few calibration tests are conducted. The TMC can shorten or extend calibration periods as needed to provide a consistent flow of reference oil test data. Adjustments to calibration periods are made such that laboratories incur no net loss or gain in calibration status.

A3.2 *Special Use of the Reference Oil Calibration System*—The surveillance panel has the option to use the reference oil system to evaluate changes that have potential impact on test severity and precision. This option is only taken when a program of donated tests is not feasible. The surveillance panel and the TMC shall develop a detailed plan for the test program. This plan requires all reference oil tests in the program to be completed as close to the same time as possible, so that no laboratory/stand calibration status is left pending for an excessive length of time. In order to maintain the integrity of the reference oil monitoring system, each reference oil test is conducted so as to be interpretable for stand calibration. To facilitate the required test scheduling, the surveillance panel may direct the TMC to lengthen and shorten reference oil calibration periods within laboratories such that the laboratories incur no net loss or gain in calibration status. To ensure accurate stand, or laboratory, or both severity assessments, conduct non reference oil tests the same as reference oil tests.

A3.3 *Donated Reference Oil Test Programs*—The surveillance panel is charged with maintaining effective reference oil test severity and precision monitoring. During times of new parts introductions, new or re-blended reference oil additions, and procedural revisions, it may be necessary to evaluate the possible effects on severity and precision levels. The surveillance panel may choose to conduct a program of donated reference oil tests in those laboratories participating in the monitoring system, in order to quantify the effect of a particular change on severity and precision. Typically, the surveillance panel requests its panel members to volunteer enough reference oil test results to create a robust data set. Broad laboratory participation is needed to provide a representative sampling of the industry. To ensure the quality of the data obtained, donated tests are conducted on calibrated test stands. The surveillance panel shall arrange an appropriate number of donated tests and ensure completion of the test program in a timely manner.

A3.4 *Intervals Between Reference Oil Tests*—Under special circumstances, such as extended downtime caused by industry wide parts or fuel shortages, the TMC may extend the intervals between reference oil tests. Such extensions shall not exceed one regular calibration period.

A3.5 *Introducing New Reference Oils*—Reference oils produce various results. When new reference oils are selected, participating laboratories will be requested to conduct their share of tests to enable the TMC to recommend industry test targets. ASTM surveillance panels require a minimum number of tests to establish the industry test targets for new reference oils.

A3.6 *TMC Information Letters*—Occasionally it is necessary to revise the test method, and notify the test laboratories of the change, prior to consideration of the revision by Subcommittee D02.B0. In such a case, the TMC issues an Information Letter. Information Letters are balloted semi annually by Subcommittee D02.B0, and subsequently by D02. By this means, the Society due process procedures are applied to these Information Letters.

A3.6.1 *Issuing Authority*—The authority to issue an Information Letter differs according to its nature. In the case of an Information Letter concerning a part number change which does not affect test results, the TMC is authorized to issue such a

letter. Long-term studies by the surveillance panel to improve the test procedure through improved operation and hardware control may result in the issuance of an Information Letter. If obvious procedural items affecting test results need immediate attention, the test sponsor and the TMC issue an Information Letter and present the background and data supporting that action to the surveillance panel for approval prior to the semiannual Subcommittee D02.B0 meeting.

A3.7 *TMC Memoranda*—In addition to the Information Letters, supplementary memoranda are issued. These are developed by the TMC and distributed to the appropriate surveillance panel and participating laboratories. They convey such information as batch approvals for test parts or materials, clarification of the test procedure, notes and suggestions of the collection and analysis of special data that the TMC may request, or for any other pertinent matters having no direct effect on the test performance, results, or precision and bias.

A4. ASTM TEST MONITORING CENTER: RELATED INFORMATION

A4.1 *New Laboratories*—Laboratories wishing to become part of the ASTM Test Monitoring System will be requested to conduct reference oil tests to ensure that the laboratory is using the proper testing techniques. Information concerning fees, laboratory inspection, reagents, testing practices, appropriate committee membership, and rater training can be obtained by contacting the TMC Director.

A4.2 *Information Letters: COTCO Approval*—Authority for the issuance of Information Letters was given by the committee on Technical Committee Operations in 1984, as follows: "COTCO recognizes that D02 has a unique and complex situation. The use of Information Letters is approved providing each letter contains a disclaimer to the affect that such has not obtained ASTM consensus. These Information Letters should be moved to such consensus as rapidly as possible."

A4.3 *Precision Data*—The TMC determines the precision of test methods by analyzing results of calibration tests conducted on reference oils. Precision data are updated regularly. Current precision data can be obtained from the TMC.

A5. REACTION VESSEL

A5.1 A diagram of the reaction vessel (ACE Glass, Inc. part number D120676)[6,7] is shown in Fig. A5.1.

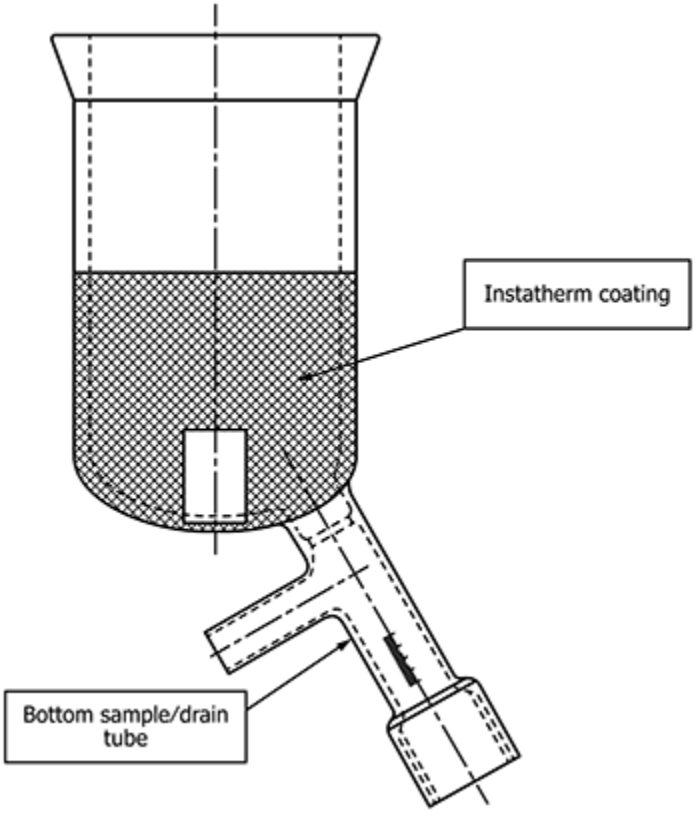

FIG. A5.1 Reaction Vessel

A6. REACTION VESSEL HEAD

A6.1 Construct the vessel head as described in Fig. A6.1. Reimel Machine, Inc. part number RMI-1002-DH[9,7] has also been shown to be suitable for this application.

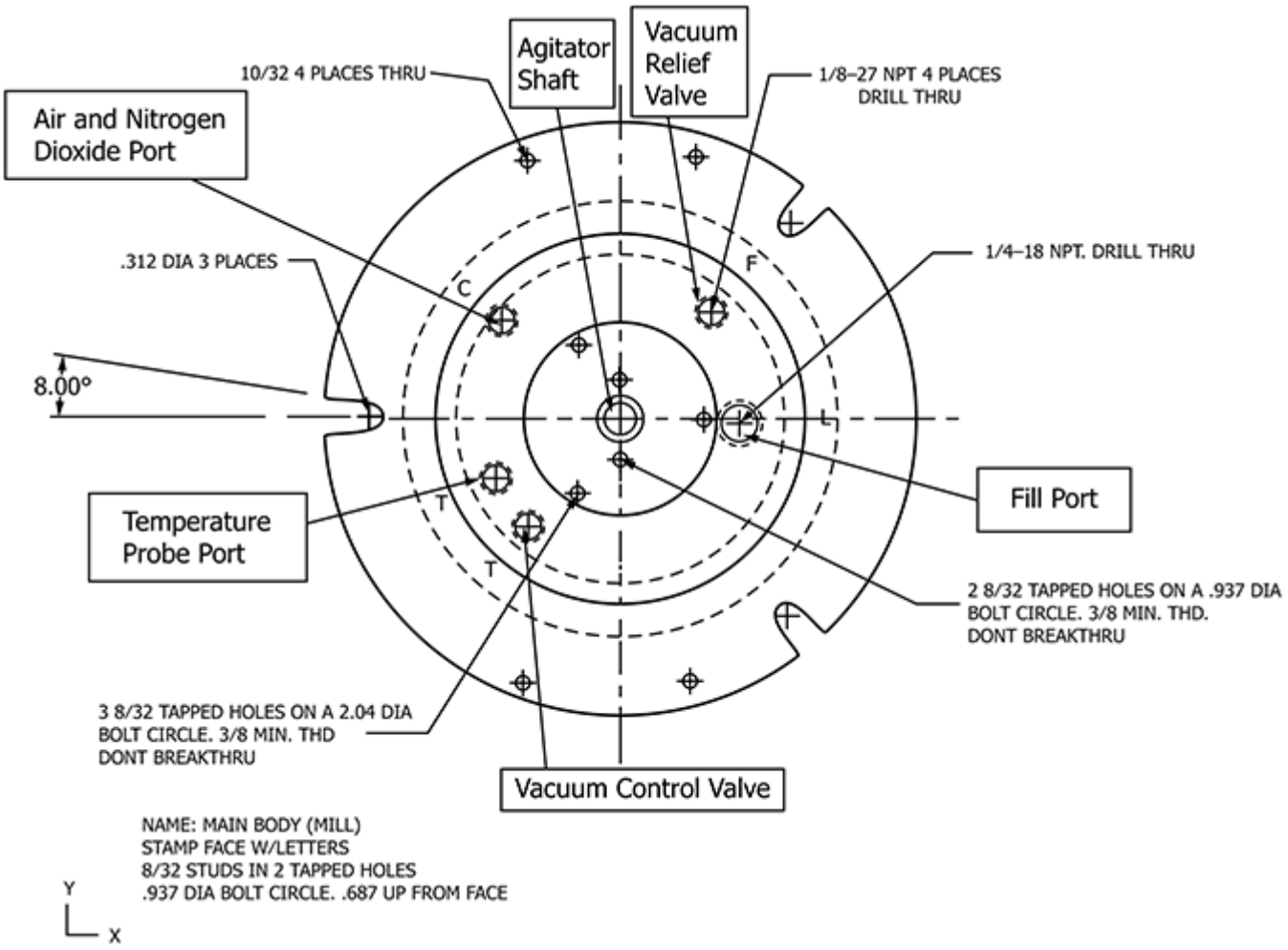

FIG. A6.1 Reaction Vessel Head (all dimensions are in inches)

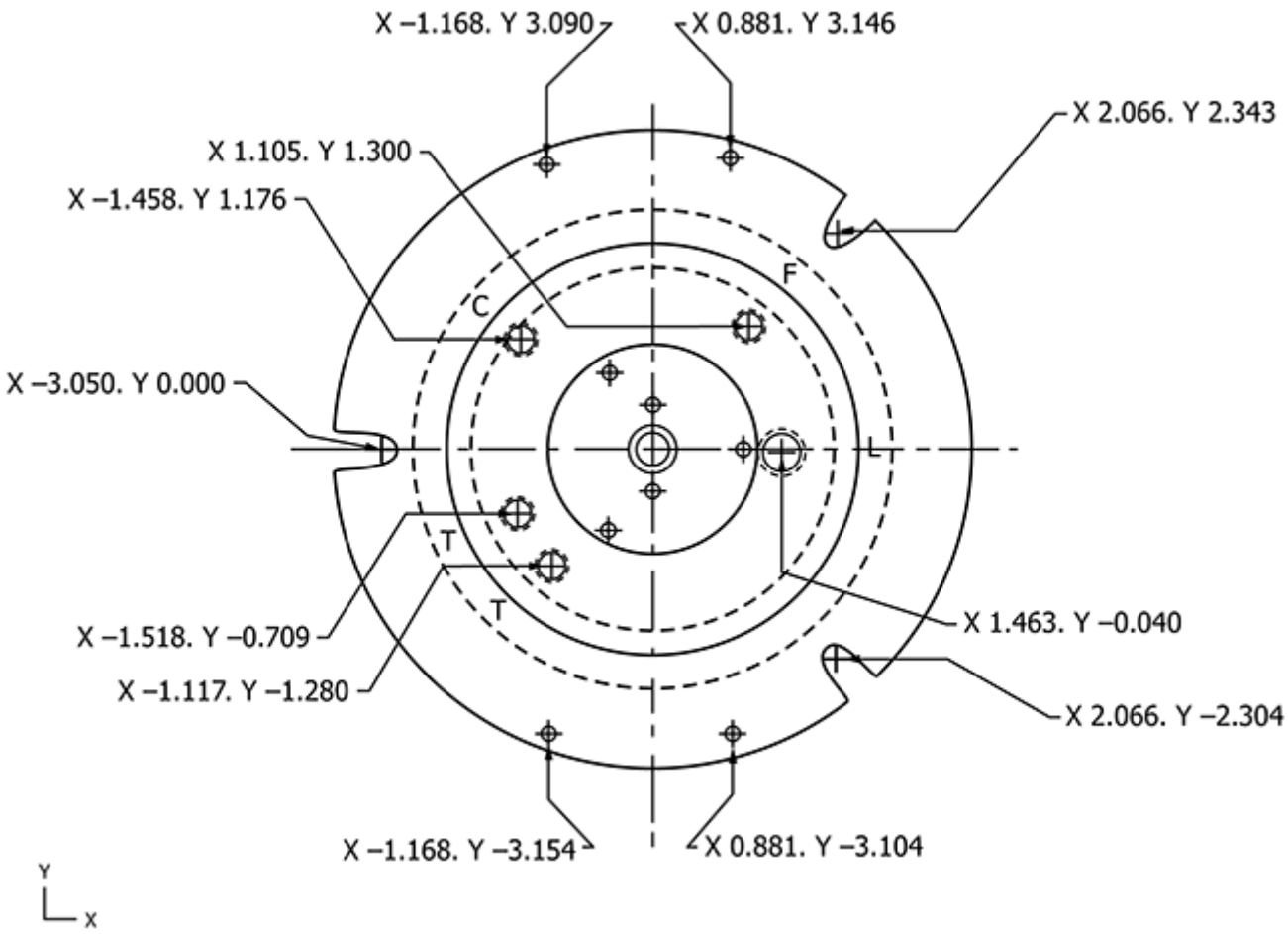

FIG. A6.1 Reaction Vessel Head (all dimensions are in inches) *(continued)*

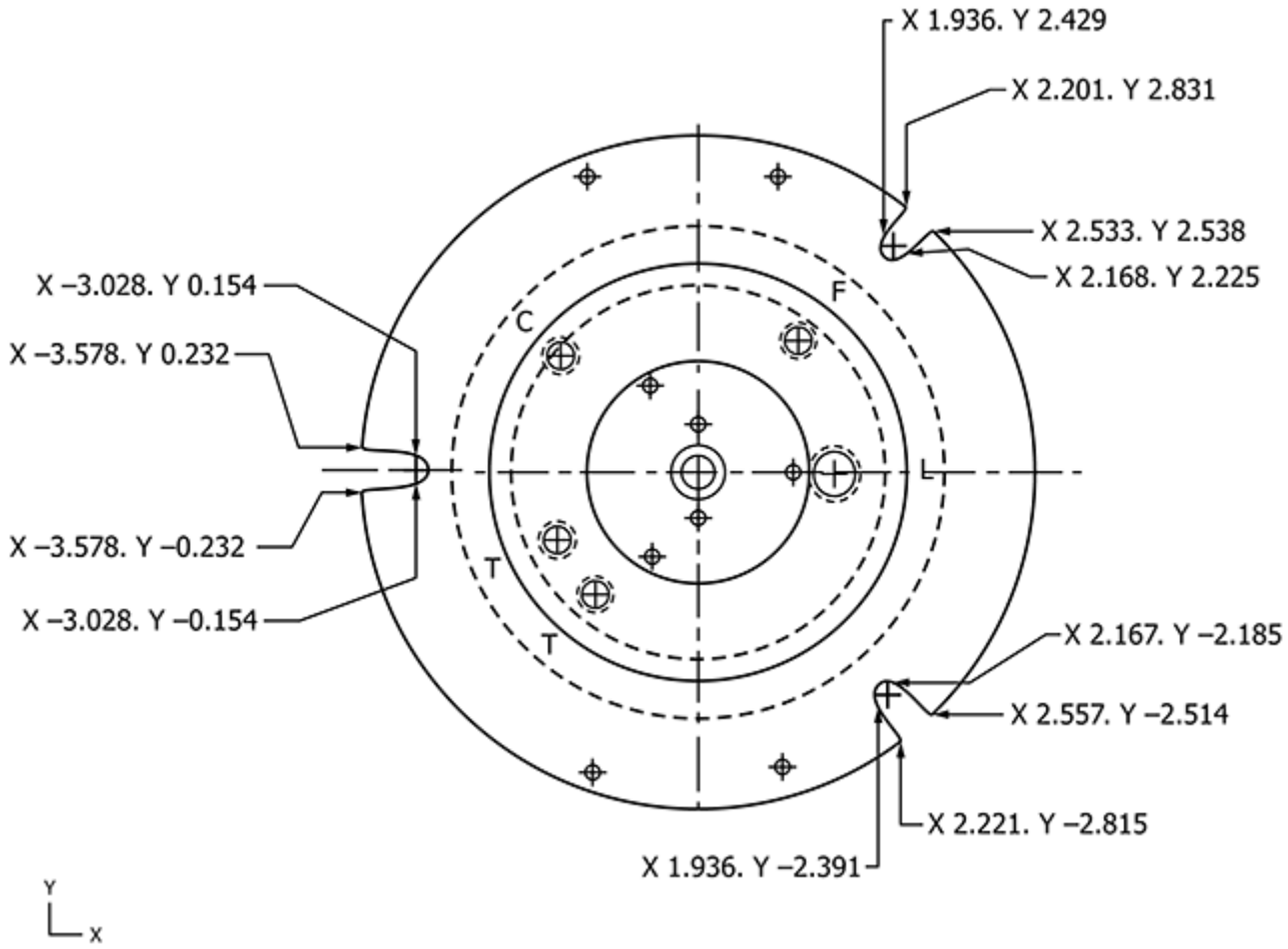

FIG. A6.1 Reaction Vessel Head (all dimensions are in inches) *(continued)*

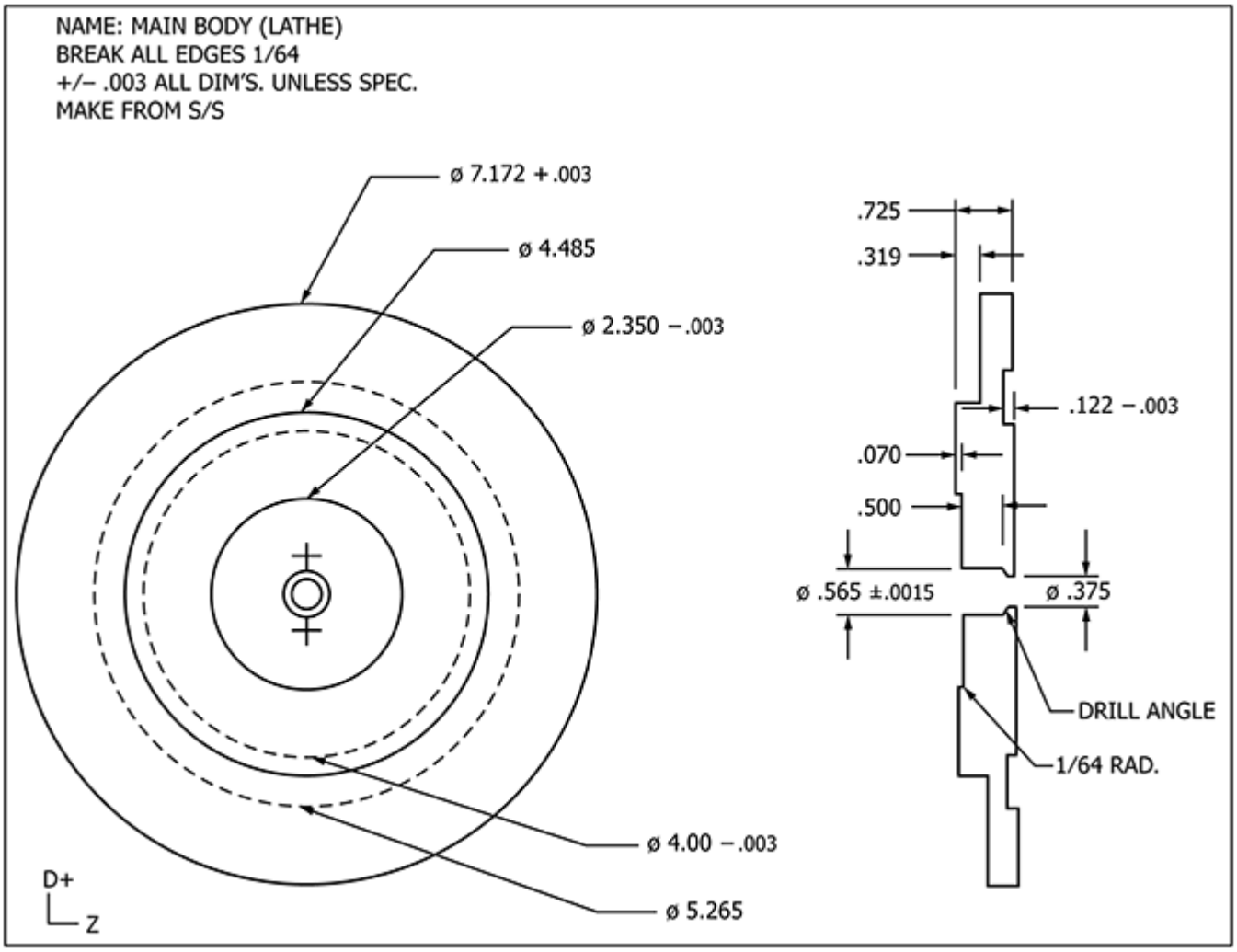

FIG. A6.1 Reaction Vessel Head (all dimensions are in inches) *(continued)*

A7. REACTION VESSEL-TO-HEAD SEAL

A7.1 Fig. A7.1 shows details and dimensions for the reaction vessel-to-head seal. Reimel Machine, Inc. part number RMI-1007-DH[9,7] has been found suitable for this purpose.

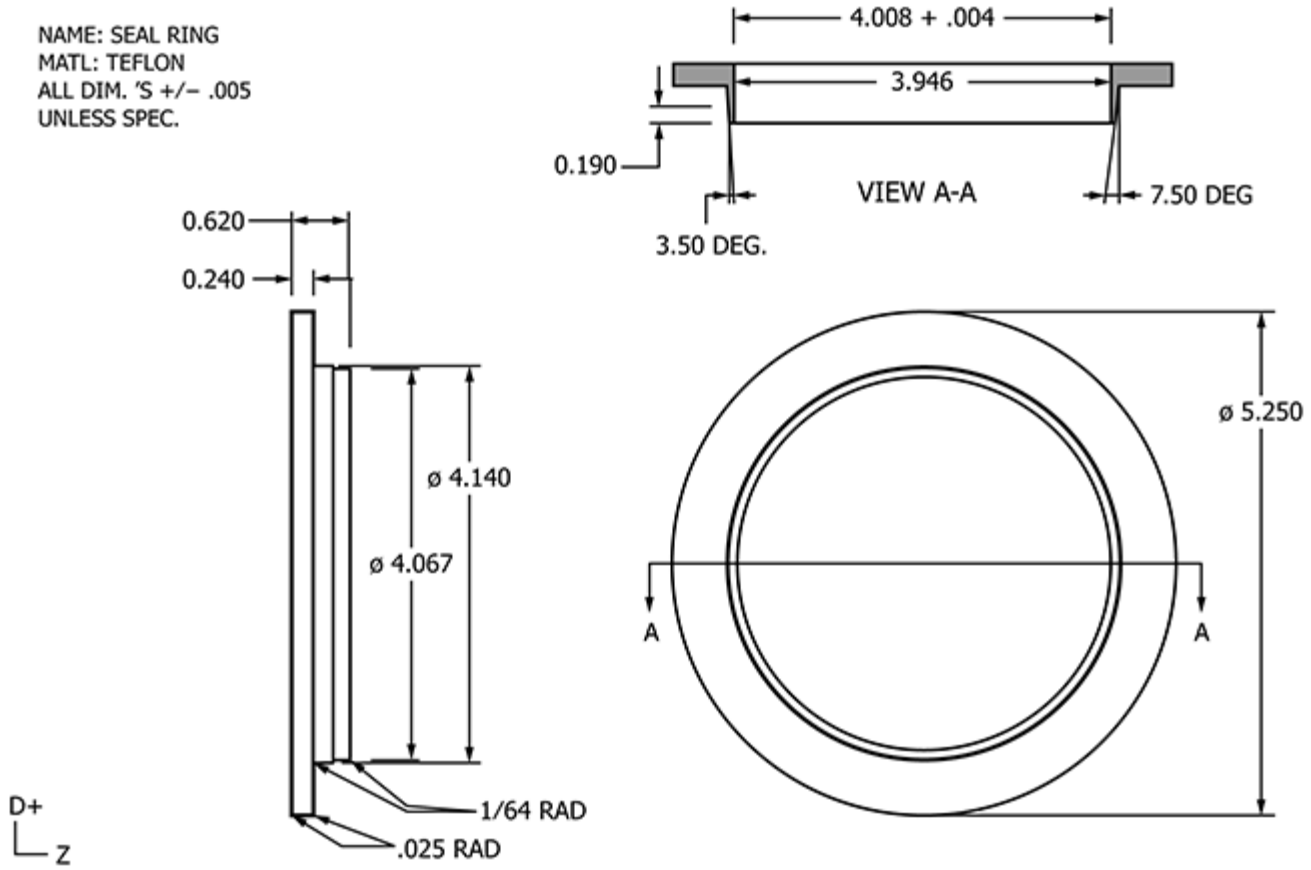

FIG. A7.1 Reactor Vessel to Head Seal (all dimensions are in inches)

A8. AGITATOR TURBINE BLADE

A8.1 Fig. A8.1 shows details and dimensions of the stainless steel agitator turbine blade. Reimel Machine, Inc. part number RMI-1001-DH[9,7] has been found suitable for this purpose.

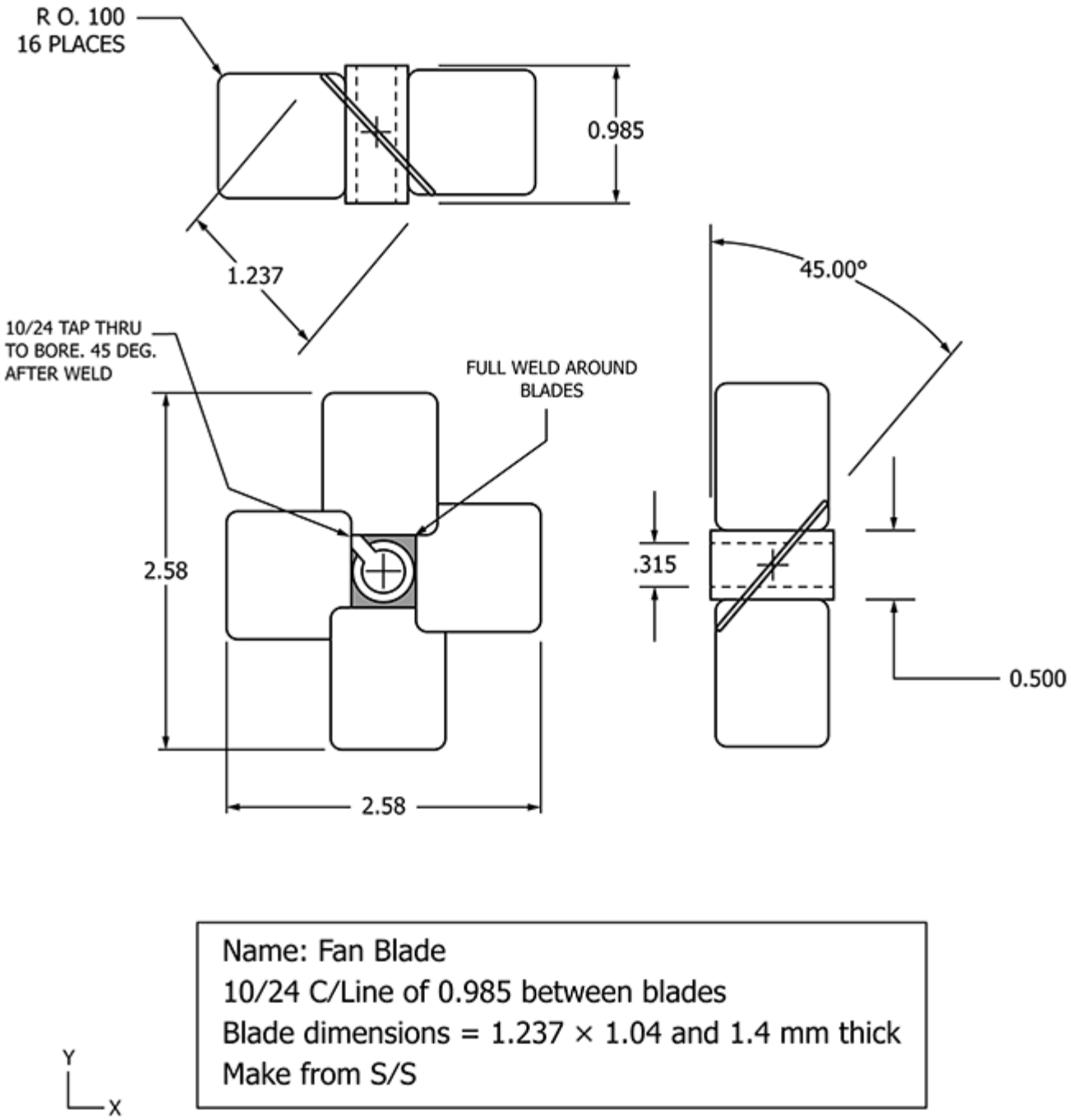

FIG. A8.1 Specification for Agitator Turbine Blades (all dimensions are in inches unless otherwise stated)

A9. AGITATOR PACKING GLAND

A9.1 Fig. A9.1 shows details and dimensions of the agitator packing gland. Reimel Machine, Inc. part number RMI-1004-DH[9,7] has been found suitable for this purpose.

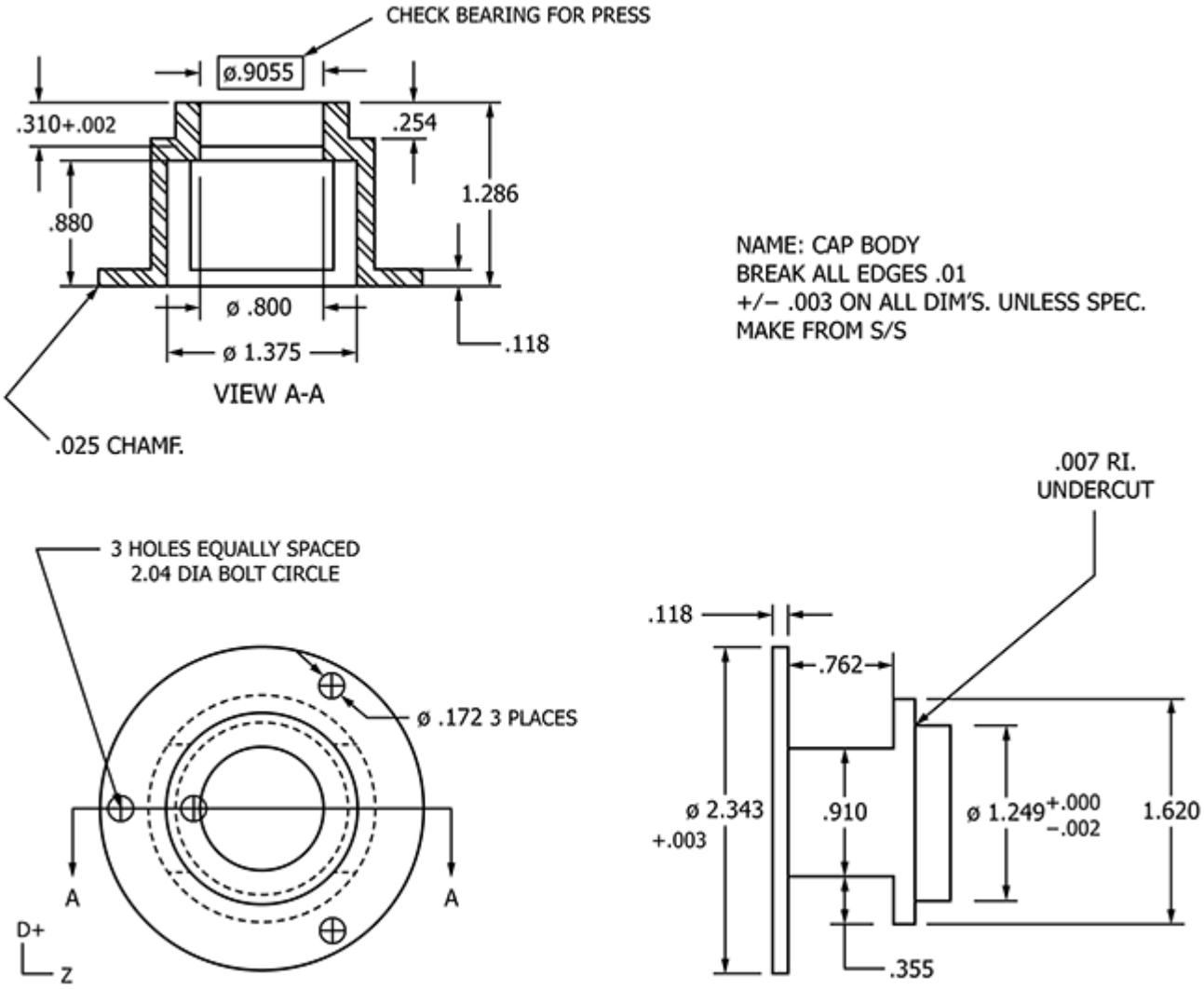

FIG. A9.1 Agitator Packing Gland (all dimensions are in inches)

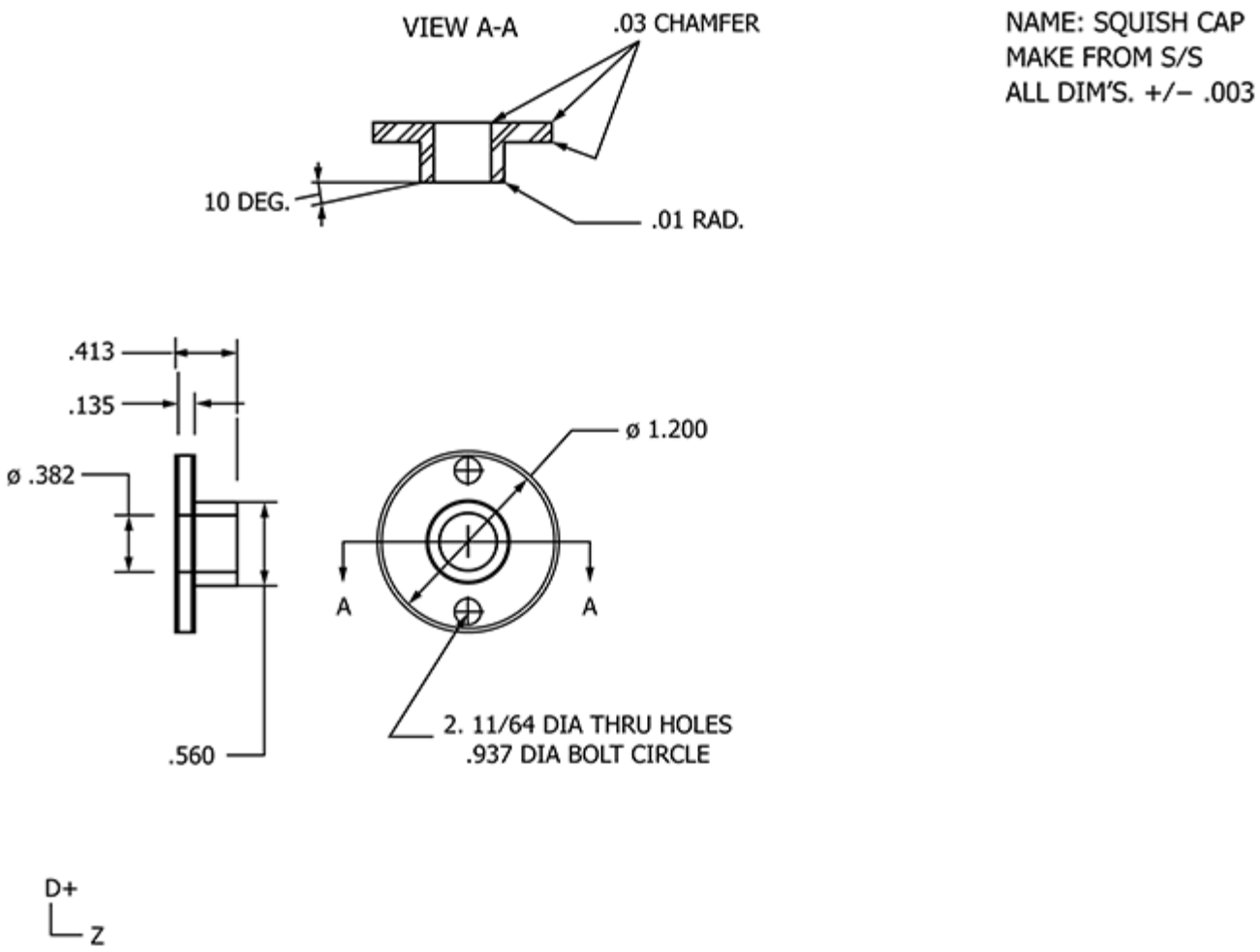

FIG. A9.1 Agitator Packing Gland (all dimensions are in inches) *(continued)*

A10. NITROGEN DIOXIDE GRADUATED TUBE

A10.1 A diagram of the Ace Glass, Inc. 12-mL, graduated centrifuge tube, part number D120677[6,7] is shown in Fig. A10.1.

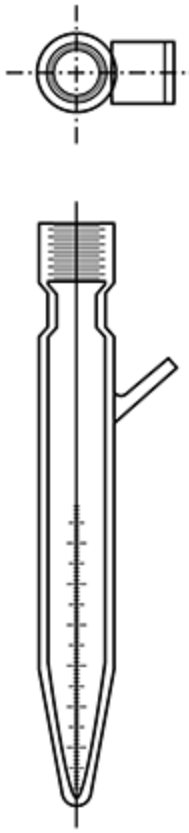

FIG. A10.1 Nitrogen Dioxide Graduated Tube

A11. VACUUM SYSTEM PLUMBING

A11.1 *General*—It is critical to follow these instructions precisely when constructing the vacuum plumbing for the reaction vessel. This is because restrictions in the vacuum system may act as a partially closed valve. As a consequence, the vacuum control valve that is used to *tune* the system, cannot be opened sufficiently to compensate and the tuning range may be inadequate to allow calibration with all reference oils in section 9.1.

A11.2 Construct vacuum lines (flexible or rigid stainless steel) from the vacuum source to condenser from tubing with a minimum inside diameter of 0.375 in. (9.53 mm). If construction is rigid tubing, use machined fittings for bends (do not use bent tubing).

A11.3 Construct vacuum lines from the condenser to the top connection of the vacuum control valve at the reactor head from tubing with a minimum inside diameter of 0.375 in. (9.53 mm) and reduced to $^{1}/_{4}$ in. tubing with inside diameter of 0.18 in. (4.6 mm) to attach onto the vacuum control valve. If construction is rigid tubing, use machined fittings for bends (do not use bent tubing). Ensure that rigid vacuum lines from the reactor to the vacuum trap condenser slope downward to the condenser and that flexible vacuum lines from the reactor to the condenser do not have any dips or constrictions.

A11.4 Construct the vacuum lines from the bottom connection of the vacuum control valve to the reactor connection from $^{1}/_{4}$ in. tubing with inside diameter of 0.18 in. (4.6 mm) and no more than 25 mm long.

A12. VACUUM TRAP CONDENSERS

A12.1 *General*—A vacuum trap/condenser is used to protect the vacuum system from harmful effects of reaction gases and to collect volatilized oil.

A12.2 A dual trap, assembled from Ace Glass, Inc. part numbers 8748-12, 7506-15, and 8751-20,[6,7] has been found to provide sufficient system protection.

A12.3 An alternative vacuum trap/condenser for $^{1}/_{2}$ in. tubing comprising an Ace Glass, Inc. condenser (part number D127507) and trap (part number D127590) has also been found to be satisfactory.[6,7] This alternative offers larger diameter connections to the vacuum system than that specified in A12.2.

A13. SETTING THE VACUUM CONTROL VALVE

A13.1 On a completely assembled reactor including the flow meter, apply vacuum to the system, open the vacuum control valve fully, and block the vacuum relief orifice long enough to assure system will attain 85 kPa with a subsurface airflow of 185 mL/min. The air flow meter shall read less than 0.6 SCFM.

A13.2 Adjust the vacuum control valve and the vacuum relief orifice to attain an airflow of 2.0 SCFM ± 0.1 SCFM through the reactor while the vacuum pressure is maintained at 61 kPa ± 1.7 kPa.

A13.3 The system is now ready to begin reference oil calibration runs as described in section 9. Do not change the vacuum control valve setting during the calibration runs as it affects the severity of the test. If the system achieves "in-calibration status" in 9.1, use the same vacuum control valve setting for all subsequent test oil runs.

A13.4 Should the system not achieve calibration in 9.1, first check that the vacuum plumbing instructions in Annex A11 were followed precisely. If they were, make the following adjustments:

A13.4.1 If a reference oil exhibits a mild response, open the vacuum control valve further. If the reference oil response is too severe, close the vacuum control valve further. Resetting the vacuum control valve is a matter of some trial and error; it is suggested that no more than one revolution of the handle be made at any one time. After resetting the vacuum control valve, adjust the vacuum relief orifice to attain an airflow of 2.0 SCFM ± 0.1 SCFM through the reactor while the vacuum pressure is maintained at 61 kPa ± 1.7 kPa.

A13.4.1.1 For systems using a laboratory vacuum pump, it may be necessary to install an air bleed to achieve the latter conditions.

APPENDIXES

(Nonmandatory Information)

X1. SAMPLE PREPARATION AND ADDITION

X1.1 *General*—There is no prescribed method of introducing the fresh oil and the sample with the catalyst into the reactor. Examples of techniques that have been successfully used are given below.

X1.1.1 *Premix Procedure:*

X1.1.1.1 Tare a clean, 300 mL beaker using the balance described in 6.1.1 and weigh 3.0 g ± 0.1 g of prepared iron ferrocene catalyst into beaker.

X1.1.1.2 Add the test oil to the beaker until a total mass of 200.0 g ± 1.0 g is attained.

X1.1.1.3 Stir catalyst/test oil mixture with a glass rod for 1 min.

X1.1.1.4 Transfer the mixture into the reaction vessel through the fill port.

X1.1.1.5 Seal the fill port with a threaded plug.

X1.1.2 *Direct Weighing Procedure:*

X1.1.2.1 Remove the reactor vessel from the apparatus, clean, if necessary, as described in 11.1 and tare using the balance described in 6.1.1.

X1.1.2.2 Weigh 3.0 g ± 0.1 g of prepared iron ferrocene catalyst into the vessel.

X1.1.2.3 Add test oil until a total mass of 200.0 g ± 1.0 g is achieved.

X1.1.2.4 Reassemble the vessel to the apparatus.

X1.1.2.5 Carry out the vessel seal check as described in 10.3.

X1.1.2.6 Preset the vacuum flow and pressure as described in 10.4.

X2. CHARGING THE LIQUID NITROGEN DIOXIDE

X2.1 *General*—There is no prescribed method of charging the liquid nitrogen dioxide. An example of a technique that has been successfully used is given below.

X2.1.1 *Using a 3-Way or a 4-Way Valve:*

X2.1.1.1 To reduce pressure inside the graduated tube, either place an ice/water mixture at the bottom of the tube or a piece of dry ice at the top of the tube.

NOTE X2.1—Nitrogen dioxide boils at 21.1 °C.

X2.1.1.2 Close all valves into the apparatus.

X2.1.1.3 Turn the selector valve so that it is pointed down towards the graduated tube in the case of a 3-way valve and towards the reaction vessel in the case of a 4-way valve.

X2.1.1.4 Open the nitrogen dioxide gas bottle valve for several seconds to allow some of the liquid phase to collect in the connecting tube above the valve.

X2.1.1.5 Securely close the nitrogen dioxide gas bottle valve and slowly open the nitrogen dioxide charging valve to allow some liquid to drip into the nitrogen dioxide graduated tube. (**Warning—**Because of the toxicity of nitrogen dioxide, exercise care when opening the charging valve.)

X2.1.1.6 Repeat the sequence of chilling, and valve opening and closing, until 2.0 mL of nitrogen dioxide is present in the

tube.

X2.1.1.7 Alternatively, the application of vacuum can also be used to charge the graduated tube.

NOTE X2.2—If using a vacuum to remove excess nitrogen dioxide due to an overcharge, exercise great care to avoid sucking out the entire charge.

X3. NITROGEN DIOXIDE PRECISION NEEDLE VALVE

X3.1 A needle valve positioned between the vessel and nitrogen dioxide graduated tube has been successfully used to control the flow of nitrogen dioxide gas into the inlet flow stream. Because of the small amount of gas required per hour, it is recommended that the valve have a low Cv for controlling the flow in a precise manner. Examples of valves that have been found satisfactory are a 10-turn, vernier-handle, needle valve with a Cv of 0.004 and a 20-turn, needle valve with a Cv of 0.019.

X4. EXAMPLE OF AN ASSEMBLED ROBO APPARATUS

X4.1 Fig. X4.1 shows an example of an assembled ROBO apparatus test stand. As pointed out in 6.19, however, there is no standard ROBO apparatus assembly. As an aid to building and setting up a new ROBO apparatus, a package of information is available on the TMC website.[2]

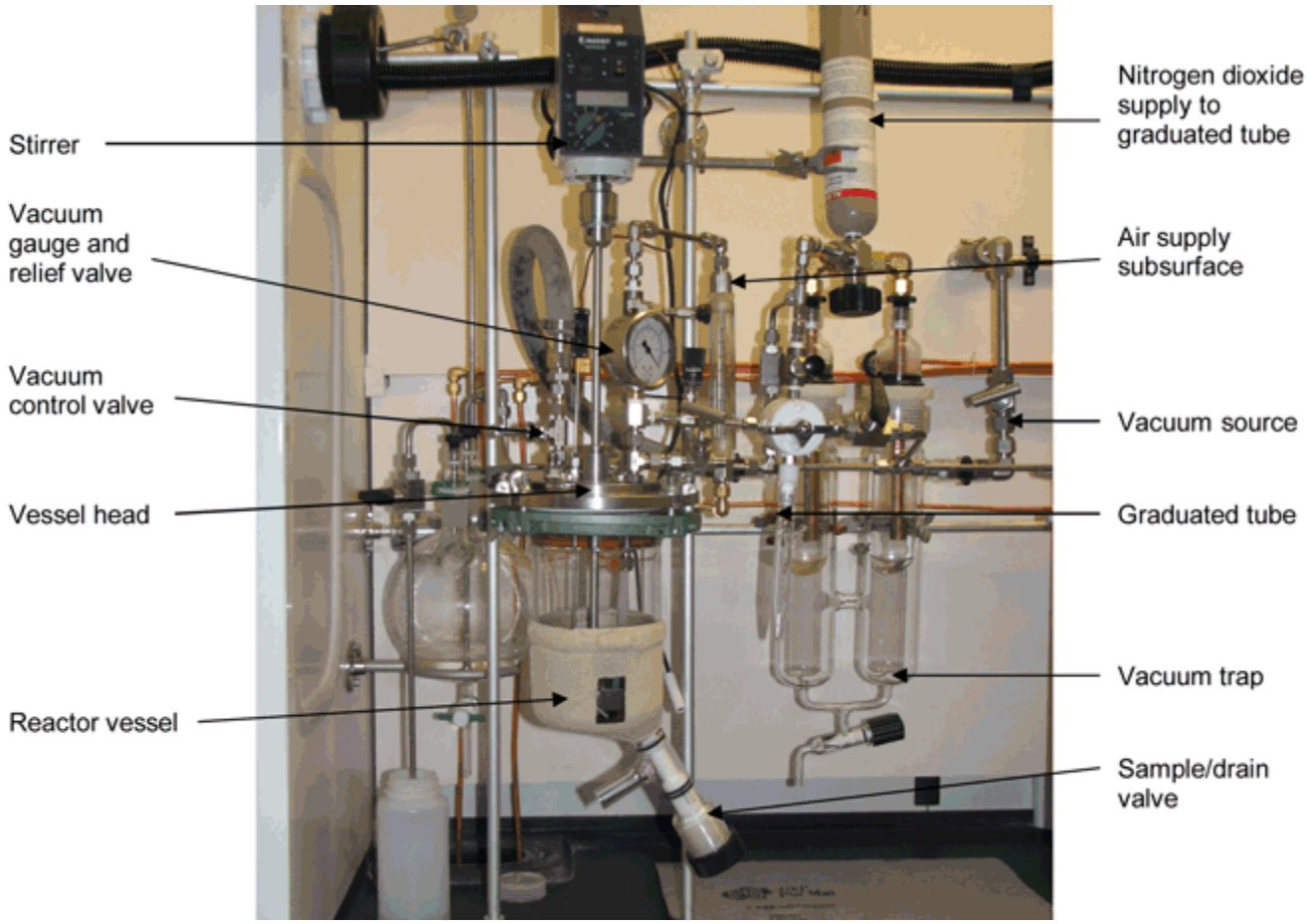

FIG. X4.1 Example of a Fully Assembled ROBO Apparatus Test Stand

X5. INFORMATION PACKAGE TO AID SETTING UP A NEW ROBO APPARATUS

X5.1 Although the information provided in ROBO Test Method D7528 is sufficient to allow a new user to build a ROBO apparatus, this package provides (non-mandatory) information, supplementary to that in Section 6 of the Method, on components and techniques that have been found suitable when setting up an apparatus for the first time. Some of the general laboratory issues associated with handling nitrogen dioxide are also discussed as are emergency shut down procedures.

X5.1.1 The information package was provided to the laboratories that built the ROBO apparatus used in the precision round

robin. Section 6 of this Method, however, incorporates improvements and clarifications of the method and takes precedent over the Information Package in the event there are discrepancies.

X5.2 The following is an index to the contents of the Information Package on TMC's website:[2]

Subject	Applicable Section of this Test Method
Background	
Agitation System	6.6
General	
Installation of the Packing Gland	
Agitation System Components	
Air Supply System	6.7
General	
Air Supply and Subsurface Feed	
Schematic of Air Supply System	
Air Supply System Components	
Miscellaneous	
General Precautions for Operating Equipment	
Emergency Shutdown Procedure	
Emergency Shutdown Procedure in the Event of a Nitrogen Dioxide Release in the Laboratory	
Nitrogen Dioxide	6.15, 10.5.3, 10.6.6
General	
Nitrogen Dioxide Physical Characteristics	
Charging the Nitrogen Dioxide to the Reaction Vessel	
Schematic	
Adjusting the Level of Nitrogen Dioxide from the Graduated Tube	
Schematic	
Nitrogen Dioxide System Components	
Temperature Control System	6.9
General	
Initial Setup Parameters	
Temperature Control System	
J-Thermocouple placement inside reactor	
Temperature Control System Components	
Vacuum System	6.10, 6.11
General	
Vacuum Trap Components	
Vacuum Trap	
Water Safety	
Vacuum Pressure Measurement	
Water Flow Diagram	
Vacuum Flow Diagram	
Vacuum System Check	
Vacuum System Components	
Vessel Head and Vessel Components	6.4
General	
Diagram and Photographs of Vessel Head	
More photographs	
Vessel and Vessel Head Components	

X6 DILUTE NITROGEN DIOXIDE IN AIR OPTION INFORMATION

X.6.1 Calculation of the necessary concentration of nitrogen dioxide in air flowing at 185 ml/min for 12.0 hours to give the same amount of nitrogen dioxide and at that same rate as in the option that uses 2.0 ml of liquid nitrogen dioxide.

Typical properties used:

Density of liquid nitrogen dioxide: 1.448 g/ml at 68F (20C)
Density of air = 1.2041 x 10^{-3} g/ml at 68F (20C)
Vapor density of nitrogen dioxide: 1.58 relative to air = 1.58 * 1.2041 * 10^{-3} g/ml = 1.903 x 10^{-3} g/ml

Volume of nitrogen dioxide in vapor phase = mass of nitrogen dioxide / vapor density of nitrogen dioxide
= (2.0 ml NO_2 x 1.448 g/ml)/(0.001903 g/ml) = 1521 ml NO_2 in vapor phase

Concentration by volume = volume of NO_2 / (volume of air + volume of NO_2)
= 100% x (1521 ml NO_2) / ((185 ml/min x 12.0 hr x 60 min/hr) + 1521 ml) = 1.129 % by volume (or rounding to 1.13 % NO_2 in air).

X6.2 Or, the amount of nitrogen dioxide added can be calculated by:
= 2.0 ml x (conc by volume NO_2 % / 1.13 %) x (time hr / 12 hr) x (time-averaged flow rate ml/min / 185 ml/min)

X7 TIME-AVERAGED SUBSURFACE AIR FLOW RATE

X7.1 If the flow rate never varies outside the range of 185 ± 2 ml/min or if a mass flow meter is used, then the deviations are deemed not significant and the time-averaged value can be estimated as either 185 or whatever value within this range the operator estimates based on the observations.
X7.2 To calculate the time-averaged flow rate, assume any changes observed in flow rate are linear between observations. If the switch from dilute nitrogen dioxide in air to dry-air is done automatically with no opportunity to observe the flow rate at that time, then calculate a flow rate by assuming that the average change in flow rate (change/hour) continues for the period between the last observation and the switching time.
X7.3 In this example, the initial flow rate is set at 185 ml/min and it decreases at 1 ml/min until the switching time at about 12 hours. One of the observation times is not exactly on the hour. The observed readings have some small variations. The switching time is not exactly at 12.0 hours. The calculations are done in a spreadsheet shown in Table X7.1

ROBO dilute NO2 flow rate

Time from start (hr)	Flow rate (ml/min)	Time since last reading (hr)	Average flow rate	Flow x Time (hr ml/min)	Change flow rate/time
0	185	0			
1	184	1	184.5	184.5	-1
1	185	0			
2.1	184	1.1	184.5	202.95	-0.9090909
2.1	185	0			
3	185	0.9	185	166.5	0
3	185	0			
4	183	1	184	184	-2
4	185	0			
5	184	1	184.5	184.5	-1
5	184	0			
6	183	1	183.5	183.5	-1
6	185	0			
12.1					

Average change flow rate/time					-0.9848485
Calculated at switch time	178.9924	6.1	181.9962	1110.1769	
Time-averaged flow rate			183.151		
If measured at switch time	179	6.1	182	1110.2	-0.9836066
Time-averaged flow rate			183.1529		
True average flow rate (with assumptions)			183.2145		

Table X7.1 Robo Dilute Flow Rate Calculation

Shaded data is input, the rest of the values are calculated. Observations are made every hour for 6 hours (in this case) and the 12 hour observation is either calculated or observed at 12.1 hours. The time since last reading is the time between observations. Two observations are recorded at each observation time, the value at the observation time and the adjusted value at that same time. Adjustments are optional. The average flow rate is the average for the time period between two observations. The Flow x Time column is the product of the time since last reading and the average flow for that time period. It is used to calculate the time-averaged flow rate. The change flow rate/time is the difference between the flow at an observation (before any adjustment) and the flow in the previous observation (after any adjustment) divided by the time between observations. It is only calculated for time intervals that are not zero. It is used to calculate the flow rate at the switching time if it not observed then.

The Average change flow rate/time is the average in that column. This assumes the change in flow rate/time is constant and the variation observed is due to measuring variability, which in this case is a good assumption. If the change flow rate/time systematically changes over time, then continued observations for the remaining time would be advised.

The calculated (flow rate) at switch time is the flow rate at the last observed time (before the switch time) plus average change flow rate/time times the time between the switch time and the preceding observation. This can be used in cases where the flow rate is not observed at the switch time. The time-averaged flow rate is then the sum of the flow x time values divided by the sum of the time since last reading values (which should equal the switch time).

If the flow rate at the switching time is observed, in this case 179 ml/min, then it does not need to be estimated and is used in the calculation. In this case, a very similar calculated result is obtained.

The value for the true average flow rate for this example is the value calculated where the flow rate changes precisely at 1.0 ml/min each hour, it is adjusted back to exactly 185 ml/min at each observation, and the switch occurs at 12.1 hours.

The flow rates will ultimately be reported to three significant figures, but more figures were used during the calculations to avoid rounding errors.

SUMMARY OF CHANGES

Subcommittee D02.B0.07 has identified the locations of selected changes to this standard since the last issue (D7528-17a) that may impact the use of this standard. (Approved xxxxxx)

An option to use dilute nitrogen dioxide in air instead of using liquid nitrogen dioxide was added. This was done with changes to sections 6.7, 6.8, 6.10, 6.15, 7.1, 9.2, 10.3, 10.5, 10.6 and 13.3 and the addition of Appendix X6 and Appendix X7.