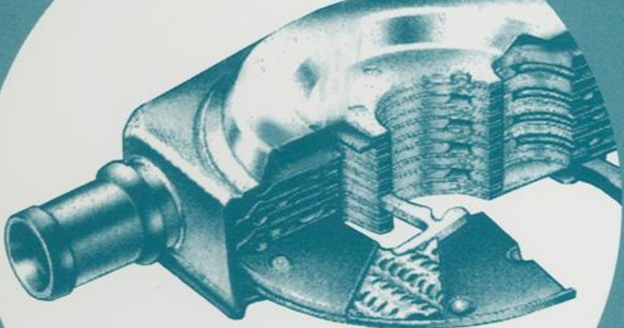
OIL FLOW STUDIES at LOW TEMPERATURES in MODERN ENGINES



HAL SHAUB
EDITOR

STP 1388

STP 1388

Oil Flow Studies at Low Temperatures in Modern Engines

Hal Shaub, editor

ASTM Stock Number: STP1388

ASTM
100 Barr Harbor Drive
PO Box C700
West Conshohocken, PA 19428-2959

Printed in the U. S. A.

Library of Congress Cataloging-in-Publication Data

Oil flow studies at low temperatures in modern engines / Hal Shaub, editor.
p. cm.—(STP; 1388)
"ASTM Stock Number: STP1388."
Proceedings of a symposium held June 21, 1999, in St. Louis, Missouri.
Includes bibliographical references.
ISBN 0-8031-2857-6
1. Internal combustion engines—Lubrication—Congresses. 2. Lubrication and lubricants—Congresses. I. Shaub, Hal, 1935- II. American Society for Testing and Materials. III. ASTM special technical publication; 1388.

TJ789.O355 2000
621.43—dc21

00-044157

Peer Review Policy

Each paper published in this volume was evaluated by two peer reviewers and at least one editor. The authors addressed all of the reviewers' comments to the satisfaction of both the technical editor(s) and the ASTM Committee on Publications.

To make technical information available as quickly as possible, the peer-reviewed papers in this publication were prepared "camera-ready" as submitted by the authors.

The quality of the papers in this publication reflects not only the obvious efforts of the authors and the technical editor(s), but also the work of the peer reviewers. In keeping with long-standing publication practices, ASTM maintains the anonymity of the peer reviewers. The ASTM Committee on Publications acknowledges with appreciation their dedication and contribution to time and effort on behalf of ASTM.

Printed in Scranton, PA
July 2000

Foreword

This publication, *Oil Flow Studies at Low Temperatures in Modern Engines,* contains papers presented at the symposium of the same name held in St. Louis, Missouri, on June 21, 1999. ASTM Committee D02 on Petroleum Products and Lubricants sponsored the symposium. Harold (Hal) Shaub, Center for Innovation, Inc., Irving, Texas, presided as the symposium chairman and is the editor of this publication.

Contents

SESSION II

Overview

A symposium titled "Oil Flow Studies at Low Temperatures in Modern Engines" was sponsored by ASTM Committee D02 on June 21, 1999, at the Adams Mark Hotel, St. Louis, Missouri, to summarize and discuss the results of industry (ASTM and others) studies on low temperature oil flow in modern engines with modern oils. The symposium was held in conjunction with the June 20–25, 1999 Committee D02 standards development meetings. Twelve papers were presented during two (AM and PM) sessions. In addition to the studies carried out under the auspices of ASTM, the symposium provided a forum for presentation and discussion of similar studies conducted outside of ASTM technical committees. Seven papers covered the ASTM work while five papers summarized results and conclusions from the independent industry studies. The overall aim of the symposium was to assist the industry in understanding the issues involved in oil flow related engine operation at low temperatures in modern engines (lubricated with modern oils) and to provide a good basis for future specification development.

An extensive industry (ASTM) program on cold start and pumpability, with modern oils in modern engines, was designed and carried out over the past six plus years. This study was in response to four specific concerns voiced by the Fuels and Lubricants (F&L) Division of the Society of Automotive Engineers (SAE) in a May 1992 letter to ASTM Subcommittee D02.07 on Flow Properties. The SAE F&L Division pointed out that five technical changes in modern engines and oils could lead to substantial lowering of the starting temperatures of these engines which could, in turn, adversely effect low temperature pumpability. The five changes included (1) low friction engine designs, (2) fuel injection systems, (3) computer control of ignition and fuel flow, (4) higher power-density starting motors and batteries, and (5) friction modified engine oils.

A Low Temperature Engine Performance (LTEP) task force was established within ASTM Subcommittee D02.07C in June 1992 to study and address the four SAE concerns, namely:

- To determine if modern engines start (on average) at lower temperatures than earlier engine designs do.
- To determine if there is a "safety margin" between limiting cranking and pumping viscosities in modern engines.
- To determine the cranking and pumping limitations of single grade (non-VI improved) engine oils.
- To assess the benefits and limitations of current methods for identifying oils that could result in pumpability failures in engines.

The work reported at the June 21, 1999 symposium went a long way in answering the four SAE F&L Division concerns about low temperatures oil flow in modern engines with modern oils. More specifically, the studies carried out within ASTM and reported in the first seven papers of the symposium, showed that modern (1993–94 model year) engines do indeed start, on average, at substantially lower temperatures than earlier engine designs. Because of this and in order to maintain at least a 5°C safety margin between limiting cranking and pumping viscosities, the SAE, in mid 1995 lowered the MRV measuring temperatures by 5°C and raised the limiting viscosities for each SAE "W" viscosity grade. As a result, the five multigrade and one monograde oil used in the LTEP study demonstrated acceptable pumpability at temperatures 5°C to 9°C lower than the minimum starting temperatures. Although CCS viscosity correlated with engine startability in the LTEP studies, the limiting starting viscosities were considerably higher than the SAE J300 April 1997 limits. Therefore, it was recommended that the industry revisit the cold cranking viscosity/temperature limits. Recently

(Dec. 1999) SAE has successfully balloted modifications to CCS "W" grade measurement temperatures and viscosity limits which are more in line with cold starting data from the LTEP program.

The ASTM LTEP work also showed (1) under appropriate cooling and operating conditions, air-binding pumpability could be generated in certain engine types and that (2) air-binding was only observed in these engines when significant structure was detected in the oils (gel index >40, MRV yield stress 70-105 Pa). This work helped address the fourth SAE concern noted earlier; namely, "to assess the benefits and limitations of current bench test methods for identifying oils that could result in pumpability failures in engines."

Work conducted outside the ASTM LTEP Program also helped address the fourth SAE concern. A paper titled, "SAE 5W-30 Pumpability Studies in Modern 4- and 8-Cylinder Engines: Gelation Index and MRV Effects," by C. J. May, et al., showed that four oils with gelation indices (GI) ranging from 6.0 to 15.7 did not produce air-binding tendencies in full scale engines (2.0L I-4 and 4.6 L V-8) tests. Several drain samples from those pumpability tests showed reduced GI values relative to those for the fresh oils. Perhaps, even short-term engine operation can "break-up" the subtle low temperature structures detected during Scanning Brookfield testing. A second paper titled, "Relation between Low-Temperature Rheology of Lubricating Mineral Oils and Gelation Index," by R. M. Webber, et al., proposes and presents evidence to support the supposition that the gelation index characterizes the onset of nucleation rather than the formation of macroscopic wax crystals that would be associated with a yield stress and gelation.

As far as the future is concerned, used oils in general and, more specifically, sooted oils and highly oxidized oils and their influence on low temperature pumpability are rapidly becoming a new challenge. New skills and perhaps new instruments may be needed to predict the effects of these factors on low temperature pumpability. In preliminary work, described in a paper by T. W. Selby titled, "Pumpability - Past Accomplishments; Present and Future Challenges," a new bench test technique [The Scanning Brookfield Technique-Extended Range (SBT-XR)] has shown promise in predicting low temperature pumpability with highly oxidized (thickened) and sooted used oils. A task force has already been formed in ASTM Subcommittee D02.07 to address these future needs and to investigate the SBT-XR and other approaches as potential viable predictors of "used oil" pumpability.

Much thanks goes to Dr. K. O. Henderson of Cannon Instrument Company, then chairman of ASTM Subcommittee D02.07, for suggesting that this symposium be held; Dr. C. J. May of Imperial Oil Ltd., chairman of the LTEP task force, organizer of the ASTM presentations at this symposium, for acting as symposium moderator, and for the symposium paper authors and ASTM personnel that have made this publication possible.

H. Shaub
Center for Innovation, Inc.,
Irving, TX 75038
Vice Chairman, Subcommittee D02.07
Symposium Chairman and Editor

Session I

Christopher J. May,[1] Enrique F. De Paz,[2] Fred W. Girshick,[3] Kenneth O. Henderson,[4] Robert B. Rhodes,[5] Spyros Tseregounis,[6] and Lisa H. Ying[7]

Cold Starting and Pumpability Studies in Modern Engines - Results from the ASTM D02.07C Low Temperature Engine Performance Task Force Activities: Background and Organization

Reference: May, C. J., De Paz, E. F., Girshick, F. W., Henderson, K. O., Rhodes, R. B., Tseregounis, S., and Ying, L. H., **"Cold Starting and Pumpability Studies in Modern Engines - Results from the ASTM D02.07C Low Temperature Engine Performance Task Force Activities: Background and Organization,"** *Oil Flow Studies at Low Temperatures in Modern Engines, ASTM STP 1388*, H. Shaub, Ed., American Society for Testing and Materials, West Conshohocken, PA, 2000.

Abstract: In response to a request by SAE, the Low Temperature Engine Performance (LTEP) task force was established within Section 7C in 1992 to determine the starting and pumping characteristics of modern engines using multigrade and single-grade oils, determine if there existed a "safety margin" between limiting cranking and pumping viscosities in modern engines, and assess the benefits or limitations of current lab tests for identifying oils which could result in pumpability failures in engines. This paper discusses the background surrounding the activity and the organization of the task force.

Keywords: cold starting, pumpability, lubricant rheology, light duty engines

Background

In May 1992, the Society of Automotive Engineers' Fuels and Lubricants Division sent a request to ASTM asking that Subcommittee D02.07 and its appropriate task forces organize and conduct a low-temperature engine test program [*1*]. The last ASTM engine pumpability studies had been conducted almost 20 years before [*2*], with industry cold

[1] Senior Research Associate, Imperial Oil, Research Dept., P.O. Box 3022, Sarnia, Ont., Canada N7T 8C8.
[2] Project Leader, Consumers Union, 101 Truman Avenue, Yonkers, NY 10703-1057.
[3] HD Formulation Science Leader, Infineum USA LP, P.O. Box 536, Linden, NJ 07036.
[4] Vice President of Technology, Cannon Instrument Co., P.O. Box 16, State College, PA 16804-0016.
[5] Consultant, Infineum, USA LP, 11735 Normont Dr., Houston, TX 77070.
[6] Staff Research Engineer, General Motors Research Laboratories, P.O. Box 9055, Warren, MI 48090-9055.
[7] Senior Statistician, Infineum USA LP, P.O. Box 536, Linden, NJ 07036.

cranking work done before that [*3-5*]. It should be noted that many studies have reported on various aspects of cold starting, pumpability and correlation to laboratory methods since the last ASTM studies [*6-44*]. However, by the early 1990s, independent studies were published which suggested that the safety margin between cold starting (cranking) and pumpability which was built into the SAE J-300 specification might not be valid for modern engines [*37-39*]. In his May 1992 letter [*1*], Sheahan cited five changes in engines and lubricants which could be lowering the starting temperatures of these engines including low-friction engine designs, fuel injection systems, computer control of fuel flow and ignition, high power-density starting motors and batteries, and friction-modified engine oils. SAE specifically asked ASTM to address four goals:

(1) to determine if modern engines start (on average) at lower temperatures than have earlier engine designs,
(2) to determine if there exists a "safety margin" between limiting cranking and pumping viscosities in modern engines,
(3) to determine the cranking and pumping limitations of single-grade (non VI-improved) engine oils, and
(4) to assess the benefits or limitations of current methods for identifying oils which could result in pumpability failures in engines.

In response to this request, the Low Temperature Engine Performance task force was established within Section D02.07C in June 1992, and has been actively investigating these issues. More than twenty task force meetings were held through December 1998, with progress reports made to the SAE Engine Oil Viscosity Classification task force and to the Engine Manufacturer's Association during that time.

The scope and objectives of the task force were established to align with SAE concerns, i.e., for both light-duty and heavy-duty engines:

- determine the starting and pumping characteristics of modern engines using multigrade and single grade oils,
- determine if there exists a "safety margin" between limiting cranking and pumping viscosities in modern engines,
- assess the benefits or limitations of current lab tests for identifying oils which could result in pumpability failures in engines.

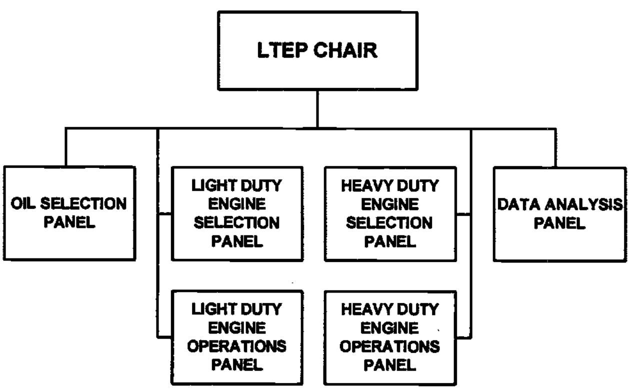


Figure 1 - *LTEP Organizational Structure*

Table 1 - *ASTM D02.07C LTEP Task Force Membership (1992-1998)****

D.L. Alexander, Alexander Technologies	R. Hoover, Cannon Instrument Co.
K. Blalock, Lubrizol Corp. [1]	A.J. Lonardo, Exxon Chemical Co. [1]
C. Bongard, K.O. Henderson, Castrol, Inc.	R.E. Manning, Consultant
A.J. Brunett, SouthWest Research Inst. [2]	P. Maggi, Cannon Instrument Co.
P.J. Burnett, Shell Development Co.	C.J. May, Imperial Oil [5,6,9]
W.A. Buscher, Jr., Texaco Inc.	S. Miller, Shell Canada Ltd.
W. Cave, John Deere PEC	A. Osio, PDVSD, Intevep
B. Clampitt, Pennzoil	C. Passut, Ethyl Corp.
E. De Paz, Texaco Inc. [9]	B.L. Papke, Equilon Enterprises, LLC
D. Deckman, Mobil R&D	R.J. Patrick, Citgo Petroleum Corp.
M. Devlin, Ethyl Corp.	R.B. Rhodes, Shell Development Co. [7,9]
K. Eiden, Chevron Research	K. Schriewer, Ford Motor Co.
R. Farina, Chevron Research	T.W. Selby, Savant, Inc.
R.L. Freerks, Chevron Chemical [3]	H. Shaub, Slick 50 Corp.
A. Gauthier, Elf Antar	J.L. Smith, Texaco Inc.
H.F. George, Lubrizol Corp.	R. Stambaugh, RohMax
F.W. Girshick, Exxon Chemical Co. [9]	J. Taylor, Ravenfield Designs Ltd.
J. Graham, Cummins Engine Co.	S. Tseregounis, General Motors R&D [9]
T.E. Hayden, Texaco Inc.	M. Wysocki, Pennzoil Products Co.
K.O. Henderson, Castrol, Inc. [4,9]	L. Ying, Exxon Chemical Co. [8,9]
V.L. Higgins, Shell Chemical Co.	

(1) Past chair, Data Analysis Panel
(2) Chair, Heavy Duty Engine Operations Panel
(3) Chair, Heavy Duty Engine Selection Panel
(4) Past chair, task force
(5) Chair, task force
(6) Chair, Light Duty Engine Operations Panel and Engine Selection Panel
(7) Chair, Oil Selection Panel
(8) Chair, Data Analysis Panel
(9) Research Report Working Group Member
*** Participants' affiliations indicated here were those at the time that the majority of task force work was done. Some member's affiliations have since changed.

Organization

The task force was organized into panels, each addressing an aspect of the planned work (Figure 1). Separate engine selection and engine operations panels were established for light-duty and heavy-duty engines, while an oil selection panel and data analysis panel handled test oil issues and analysis of the results, respectively. Task force membership included a wide range of original equipment manufacturers, oil and additive manufactures; Table 1 covers the task force membership through December 1998. Work conducted under the auspices of this task force was funded by individual participants.

It should be noted that while oil selection and test protocols were developed with both light-duty and heavy-duty engines in mind, only light-duty engine studies have been undertaken to date, due to limited interest/support for heavy-duty engine studies.

Acknowledgements

Vehicle testing at Imperial Oil's Research Labs was supported by funding from Imperial Oil, Exxon Research & Engineering, Paramins and Castrol Inc. Imperial Oil gratefully acknowledges the loan of two vehicles by General Motors Research for cold start and limited pumpability testing, and Chrysler Engineering for the loan of one vehicle in the cold start testing program.

Engine/vehicle testing at Shell Westhollow Technology Center was supported by funding from Shell Chemical Co.

Engine/vehicle testing at Texaco R&D was supported by funding from Texaco Inc.

Cold start engine testing at SouthWest Research Institute was internally funded. Engine pumpability testing at this lab was funded by Castrol Inc.

References

[1] Sheahan, T. J., Letter to Z. M. Holubec (Chairman, ASTM Subcommittee D02.07) and F.R. Duffey (Chairman, ASTM Subcommittee D02.0B), May 29, 1992.

[2] *Low Temperature Pumpability Characteristics of Engine Oils in Full Scale Engines, ASTM DS-57*, American Society for Testing and Materials, Philadelphia, PA 19103, September 1975.

[3] Selby, T. W., "A Comparison of the Effects of Cranking Speed and Oil Viscosity on Low-Temperature Engine Starting," Automotive Engineering Congress, January 13-17, 1964, 805C.

[4] *Evaluation of Laboratory Viscometers for Predicting Cranking Characteristics of Engine Oils at 0°F and -20°F, CRC Report No. 409*, Coordinating Research Council, Inc., 30 Rockefeller Plaza, NY, 10020, April 1968.

[5] Williams, C. G., "Low Temperature Viscosity of Engine Lubricants, Using the Cold Cranking Simulator, and Engine Cranking Studies," Report of the sub-Committee on Low Temperature Characteristics of Engine Lubricants, June 1970.

[6] Smith Jr., M. F. and Graham, J. P., "Pumpability of Multigrade Engine Oils at Low Temperature," SAE Paper No. 710139.

[7] McMillan, M. L., Stewart, R. M., Smith, Jr., M. F. and Rein, S. W., "Low Temperature Engine Oil Pumpability in Full-Scale Engines," SAE Paper No. 750691.

[8] Stewart, R. M., "Engine Pumpability and Crankability Tests on Commercial "W" Graded Engine Oils Compared to Bench Test Results," SAE 780369.

[9] Caracciolo, F. and McMillan, M. L., "Effect of Engine Oil Viscosity on Low-Temperature Cranking, Starting and Fuel Economy." SAE Paper No. 790728.

[10] Eberan-Eberhorst, C. G. A., Neveu, C., Rossi, A. and Thompson, M., "Low Temperature Starting and Pumpability Requirements of European Automotive Engines," SAE Paper No. 790727.
[11] Stover, W. H., "Cold Starting and Oil Pumpability - an Evaluation of New and Used Oils in Gasoline Engines." SAE Paper No. 790729.
[12] Shaub, H., Smith, M. F. and Murphy, C. K., "Predicting Low Temperature Engine Oil Pumpability with the Mini-Rotary Viscometer," SAE Paper No. 790732.
[13] Selby, T. W. and Piasecki, D. A., "Low Shear, Low Temperature Properties of Engine Oils - Techniques with the Brookfield Viscometer," SAE Paper No. 800367.
[14] Stambaugh, R. L., and O'Mara, J. H., "Low Temperature Flow Properties of Engine Oils," SAE Paper No. 821247.
[15] Smith, Jr., M. F., "Better Prediction of Engine Oil Pumpability Through a More Effective MRV Cooling Cycle," SAE Paper No. 831714.
[16] Stover, W. H., Thompson, W. R. and Smith, J. M., "Cold Starting and Oil Pumpability - Field Evaluations with Petroleum and Synthetic Engine Oils," SAE Paper No. 841013.
[17] Johnson, R. S., "A Laboratory Engine Test Study of Motor Oil Flow Properties in Winter Service," SAE Paper No. 841387.
[18] Stambaugh, R., "Low Temperature Pumpability of Engine Oils," SAE Paper No. 841388.
[19] Henderson, K. O., Manning, R. E., May, C. J. and Rhodes, R. B., "New Mini-Rotary Viscometer Temperature Profiles That Predict Engine Oil Pumpability," SAE Paper No. 850443.
[20] May, C. J., Habeeb, J. J. and White, A. M., "Engine Oil Low Temperature Pumpability: A Comparison of SAE 10W30, 5W30 and 0W30 Multigrade Engine Oils," Lubrication Engineering, 43, 7, 557, 1987.
[21] MacAlpine, G. A. and May, C. J., "Compositional Effects on the Low Temperature Pumpability of Engine Oils," SAE Paper No. 870404.
[22] Kopko, R. J. and Machleder, W. H., "Low Temperature Pumpability Studies in a Heavy Duty Diesel Engine," NPRA Paper FL-88-116, 1988.
[23] May, C. J. and Habeeb, J. J., "Lubricant Low Temperature Pumpability Studies - Oil Formulation and Engine Hardware Effects," SAE Paper No. 890037.
[24] Machleder, W. H. and Kopko, R. J., "Low Temperature Pumpability Studies in a Heavy Duty Diesel Engine - Part II," SAE Paper No. 890052.
[25] Stambaugh, R. L., Machleder, W. H. and Kopko, R. J., "Low Temperature Pumpability Studies in a Heavy Duty Diesel Engine - Part III," SAE Paper No. 890240.
[26] Frame, E. A., Montemayor, A. F. and Owens, E. C., "Low Temperature Pumpability of U.S. Army Diesel Engine Oils," SAE Paper No. 892053.
[27] Stambaugh, R. L., Machleder, W. H. and Kopko, R. J., "Heavy-Duty Diesel Engine Oil Pumpability at Low Temperature - A Study of Key Variables", Proceedings of the Japan International Tribology Conference, Nagoya, Japan 1990, pg 2019-2024.
[28] Henderson, K. O. and Sicking, J. M., "The Effect of Shear Rate and Shear Stress on Viscosity Determinations at Low Temperatures for Engine Oils" SAE Paper No.

902091.
[29] Smith, Jr., M. F., "Understanding Diesel Engine Lubrication at Low Temperature," SAE Paper No. 902177.
[30] Selby, T. W., "The Use of the Scanning Brookfield Technique to Study the Critical Degree of Gelation of Lubricants at Low Temperatures," SAE Paper No. 910746.
[31] Alexander, A. G., May, C. J. and Smith, C. R., "Factors Affecting Pumpability in Heavy Duty Diesel Truck Engines at Low Ambient Temperatures," SAE Paper No. 912337.
[32] Freerks, R. L. and Kahn, M. J., "Correlation Between Field and Laboratory Engine Oil Pumpability Testing in Heavy Duty Diesel Engines," SAE Paper No. 912338.
[33] Machleder, W. H. and O'Mara, J. H., "Low Temperature Pumpability Studies in a Heavy Duty Diesel Engine - Part IV," SAE Paper No. 920022.
[34] Alexander, A. G., May, C. J. and Smith, C. R., "Factors Affecting Pumpability in Heavy Duty Diesel Truck Engines at Low Ambient Temperatures - Part II," SAE Paper No. 920023.
[35] Henderson, K. O., "Low Temperature Performance of Commercial SAE 5W-30 Oils in Engines and Its Correlation with Bench Tests," SAE Paper No. 920560.
[36] Selby, T. W., "Problems in Bench Test Prediction of Engine Oil Performance at Low Temperature," SAE Paper No. 922287.
[37] Alexander, D. L., "Proposal for an ASTM Investigation of the Low-Temperature Cranking and Pumping Viscosity Requirements of Light-Duty Gasoline and Heavy-Duty Diesel Engines," ASTM D02 Subcommittee 7 meeting June 24, 1991.
[38] Spearot, J. A., "Engine Oil Viscosity Classification Low-Temperature Requirements Current Status and Future Needs," ASTM STP 1143, R.B. Rhodes, Ed., American Society for Testing and Materials, 1992.
[39] Alexander, A. G., MacAlpine, G. A., May, C. J. and Smith, C. R., "Passenger Car and Heavy Truck Cold Cranking Studies - Effects of Oil Viscosity and Vehicle Type," ASTM STP 1143, 1992.
[40] Henderson, K. O., "Pumping Viscosity by Mini-rotary Viscometer - Critical Aspects," ASTM STP 1143, 1992.
[41] Selby, T. W., "The Scanning Brookfield Technique of Low-Temperature, Low-Shear Rheology - Its Inception, Development, and Applications," ASTM STP 1143, 1992.
[42] Alexander, D. L., "Shear Stress Imposed on Oils During Low-Temperature Starting," ASTM STP 143, 1992.
[43] Machleder, W. H., O'Mara, J., Kopko, R. J. and Stambaugh, R. L., "Low-Temperature Pumpability in Gasoline Engines: Establishing Performance at Critical Locations," ASTM 1143, 1992.
[44] Alexander, D. L., "A Critical Assessment of the Low-Temperature Viscosity Requirements of SAE J300 and Its Future," SAE Paper No. 932794.
[45] *Cold Starting and Pumpability Studies in Modern Engines, ASTM Research Report RR-D02-1442*, November, 1998.

Christopher J. May,[1] Enrique F. De Paz,[2] Fred W. Girshick,[3] Kenneth O. Henderson,[4] Robert B. Rhodes,[5] Spyros Tseregounis,[6] and Lisa H. Ying[7]

Cold Starting and Pumpability Studies in Modern Engines - Results from the ASTM D02.07C Low Temperature Engine Performance Task Force Activities: Engine Selection and Testing Protocol

---

Reference: May, C. J., De Paz, E. F., Girshick, F. W., Henderson, K. O., Rhodes, R. B., Tseregounis, S., and Ying, L. H., "**Cold Starting and Pumpability Studies in Modern Engines - Results from the ASTM D02.07C Low Temperature Engine Performance Task Force Activities: Engine Selection and Testing Protocol,**" *Oil Flow Studies at Low Temperatures in Modern Engines, ASTM STP 1388*, H. Shaub, Ed., American Society for Testing and Materials, West Conshohocken, PA, 2000.

Abstract: As part of the Low Temperature Engine Performance task forces activities, letters were sent to all major light- and heavy-duty automotive manufactures to solicit input for engine selection. On the basis of these responses, and considering availability, annual production, potential sponsorship and range of pumping characteristics, 9 light duty engines (including 4-, 6- and 8-cylinder designs) were selected for further study. At the same time, protocols for testing the engines under both cold start and pumpability conditions were developed by consensus. Cold start testing called for good winter grade fuel, booster batteries and fresh engine tune-up to maximize starting potential. Pumpability testing was conducted via motoring of the engine to allow testing of the oil at/below the minimum starting temperatures in a repeatable fashion.

Keywords: cold starting, pumpability, lubricant rheology, light duty engines

---

[1] Senior Research Associate, Imperial Oil, Research Dept., P.O. Box 3022, Sarnia, Ont., Canada N7T 8C8.
[2] Project Leader, Consumers Union, 101 Truman Avenue, Yonkers, NY 10703-1057.
[3] HD Formulation Science Leader, Infineum USA LP, P.O. Box 536, Linden, NJ 07036.
[4] Vice President of Technology, Cannon Instrument Co., P.O. Box 16, State College, PA 16804-0016.
[5] Consultant, Infineum, USA LP, 11735 Normont Dr., Houston, TX 77070.
[6] Staff Research Engineer, General Motors Research Laboratories, P.O. Box 9055, Warren, MI 48090-9055.
[7] Senior Statistician, Infineum USA LP, P.O. Box 536, Linden, NJ 07036.

Introduction

This paper deals with the solicitation and selection of test engines for the cold starting and pumpability work carried out by the LTEP task force. Also reviewed in this paper, is the protocol for testing of the engines, as developed by the consensus of the task force.

Engine Selection

In late 1992/early 1993, letters were sent to North American, Japanese and European equipment manufacturers soliciting recommendations for light duty engines to study in cold starting/pumpability work. This was accompanied in December 1992 with a similar solicitation of North American HD manufacturers. Table 1 lists the original equipment manufacturers that were contacted.

The letter asked the equipment manufacturer to recommend up to 4 engines of interest to them from the standpoint of cold starting and pumpability. Details requested included displacement, number of cylinders, oil pump type, pick-up tube dimensions, oil height, fuel delivery system, approximate annual production, and interest in participating in the study (see Appendix C in Refence [*1*]).

Table 1 - *List of Equipment Manufacturers Contacted for Recommendations of Test Engines*

Light-Duty Engine Solicitations	
Chrysler Corp.	AB Volvo
Ford Motor Co.	BMW AG
General Motors R&D	Fiat Auto SA
Honda of America	Mercedes Benz AG
Volvo North America Corp.	Peugeot SA
	Porsche AG
Japan Automobile Manufacturers Assoc.	Renault Vehicles Industriels
	Rover Group
	Saab-Scania AB
	Volkswagen AG
Heavy-Duty Engine Solicitations	
Cummins Engine Co.	Detroit Diesel Corp.
Caterpillar Inc.	Mack Trucks

Based upon the responses received, a list of potential light duty test engines was developed which included key hardware characteristics. This was reviewed by the light duty engine selection panel, and by considering a range of expected pumping characteristics,[8] engine/vehicle availability, production volumes and potential sponsorship, a short list of recommended engines was developed from which final selections were made (Figure 1).

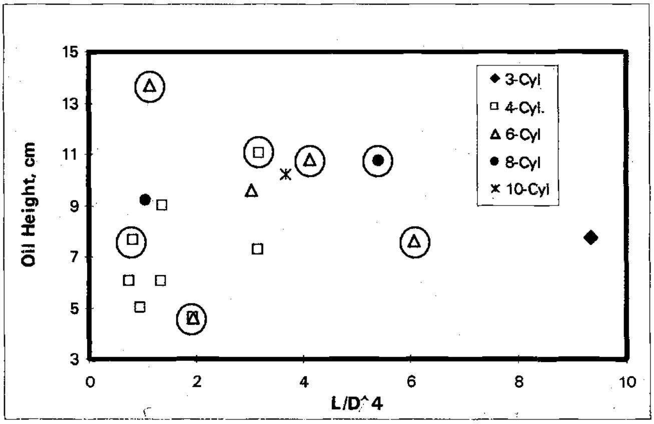


Figure 1 - *Oil Pick-up Tube Length/(Diameter^4) versus Oil Height for Recommended Engines. Circled Symbols Represent Selected Engines*

It should be noted that the selections encompassed 1993/1994 model year 4-, 6-, and 8-cylinder designs covering a substantial range of anticipated pumpability characteristics as defined by pick-up tube Length/Diameter^4 [*4*],[9] and oil height. This was considered important by the task force in order to assure a broad cross-section of designs. All engines had fuel injection delivery systems.

No testing activity in the heavy duty engine area occurred within the task force due to limited interest and lack of sponsorship from participants.

Test Protocols

Between 1993 and 1994, through a series of panel meetings, standardized protocols for conducting both light duty and heavy duty cold start and pumpability testing were developed, fine-tuned and agreed upon. The heavy duty protocol has not been used to date, due to lack of interest/sponsorship, but is included in an appendix of the LTEP research report for future use/reference (see Appendix E in Reference [*1*]). The testing protocol included details on: fired and motored tests, instrumentation/sampling, engine and oil conditioning, cooling rates, starting/running procedures, and fuel selection.

[8] Assessed by considering the oil height above the pump and the L/D^4 ratio where L = pick-up tube length and D = diameter of pick-up tube (see also [*2*], [*3*]).

[9] This analysis does not take into account any bends in the pick-up tube itself, which may significantly influence flow through the tube.

In order to meet the requirements laid out by SAE/ASTM, separate cold start and pumpability studies were undertaken on light duty vehicle/engines of interest. Fired cold start studies were conducted first, to establish minimum starting temperatures (MST) for each vehicle/oil combination. A breakdown of the participating labs and vehicles tested is included in Table 2.

Care was taken to closely control operating variables including engine state-of-tune, fuel characteristics, and battery condition to ensure adequate test precision in a "real-world" atmosphere: (i) engines were given a complete tune-up prior to testing, with

Table 2 - *Test Lab - Engine Combinations for Cold Start Testing*

Test Lab	Facility	Engine Type	Manufacturer	Model Year
Imperial Oil/Exxon	Cold Room	1) 3.3L V6 OHV 2) 2.3L I4 OHC 3) 3.8L V6 OHV 4) 1.9L I4 OHV 5) 4.6L V8 OHC 6) 3.0L V6 OHC	1) D 2) F 3) F 4) E 5) E 6) H	1) 1994 2) 1993 3) 1994 4) 1994 5) 1994 6) 1993
Shell Westhollow Tech Center	Cold Room	1) 4.0L I6 OHV 2) 3.8L V6 OHV	1) D 2) F	1) 1994 2) 1994
Texaco	Cold Box	1) 2.2L I4 OHC	1) H	1) 1994
SouthWest Research Institute	Cold Box	1) 3.0L V6 OHV	1) E	1) 1993

special attention to the fuel delivery system and throttle plate assembly, (ii) fuel distillation characteristics were selected to ensure adequate volatility, typical of current commercial practice in winter conditions in the northern U.S. and Canada,[10] (iii) the use of warm booster batteries removed a significant source of potential test variation, but also resulted in a more severe test [5-7]. It should be noted that two different labs were able to test a 3.8L V-6 from manufacturer F, to allow engine-to-engine variation to be assessed. Including these duplicates, 10 engines were used in the cold starting tests at four different labs.

Engine instrumentation allowed rpm, oil temperature and pressure to be monitored at 5 times per second during the test. The testing protocol called for conducting up to six 5-second cranking attempts for each vehicle/oil combination at a particular temperature. If cold start was achieved, the engine was shut down, the temperature lowered by 2-3°C and stabilized, then a new starting attempt was made. Once a no-start condition had been reached, the previous start temperature was recorded as the MST and then the vehicle underwent a complete fresh oil/filter change and received a new set of spark plugs, followed by repeat cold start evaluation at/below the MST achieved in the first test (see

[10] Fuel was supplied by Shell Development (for Shell testing), and Imperial Oil (for testing by Imperial Oil, SouthWest Research Institute, and Texaco). Fuel characteristics are compared in Table 3.

Table 3 - *Fuel Characteristics - Cold Start Testing (as measured by donating lab)*

Property	Imperial Oil	Shell Westhollow T.C.
T_{10}, °C	40	42.2
T5, °C	98	96.1
RVP, psi	---	10.79

Figure 2). A minimum of three complete cold starting tests on each oil/vehicle combination allowed average MSTs to be determined for greater accuracy.

Cold start testing was followed by motored pumpability testing generally conducted at/below the MSTs established in the cold start work, on a subset of engines (see Table 4 for test lab/ engine combinations); a total of 7 different engines (no duplicates) were tested by the four different labs[11]. Engine motoring was selected to be a more repeatable (but also more severe) test of pumpability than fired engine studies [*8*]. It also allowed assessment of pumpability *below* the minimum start temperatures. Motoring speed was based upon the results obtained in the cold start studies with the exception of the 2.2L I-4 (manufacturer G). Inconsistent starting speeds from the cold start work were reviewed with the OEM, who advised the lab of the designed low temperature idle speed for this engine.

Table 4 - *Test Lab - Engine Combinations for Pumpability Testing*

Test Lab/Sponsor	Facility	Engine/Manufacturer	Comments
Shell Westhollow Technology Center	Cold Room	1) 4.0L I6 / D 2) 3.8L V6 / F	Pumpability studies on LTEP 1-7 at multiple temperatures. Additional pumpability studies on LTEP 22-27 under rapid and modified cooling cycles.
Texaco	Cold Box	1) 2.2L I4 / G	Pumpability studies on LTEP 1-7 at multiple temperatures. Additional pumpability studies on LTEP 22-27 under rapid and modified cooling cycles.
SwRI/Castrol	Cold Box	1) 4.6L V8 / E	Pumpability studies on LTEP 1-7 at multiple temperatures. Additional pumpability studies on LTEP 22-27 under rapid and modified cooling cycles.
Imperial Oil/Exxon	Cold Room	1) 2.3L I4 / F 2) 1.9L I4 /E 3) 3.0L V6 / H	Pumpability testing limited to LTEP 1-5 at minimum start temperatures (approximate), plus rapid and modified slow cool pumpability on LTEP 22

Engine instrumentation included oil temperatures (4 pan locations), oil pressure (before the pump, before/after the filter, at a main gallery (2 in V-configuration engines), and at the most remote oiling location). Data acquisition rates were 2 times per second.

[11] Due to funding limitations, only 3 labs conducted the full pumpability testing protocol on LTEP 1-7, in 4 engines.

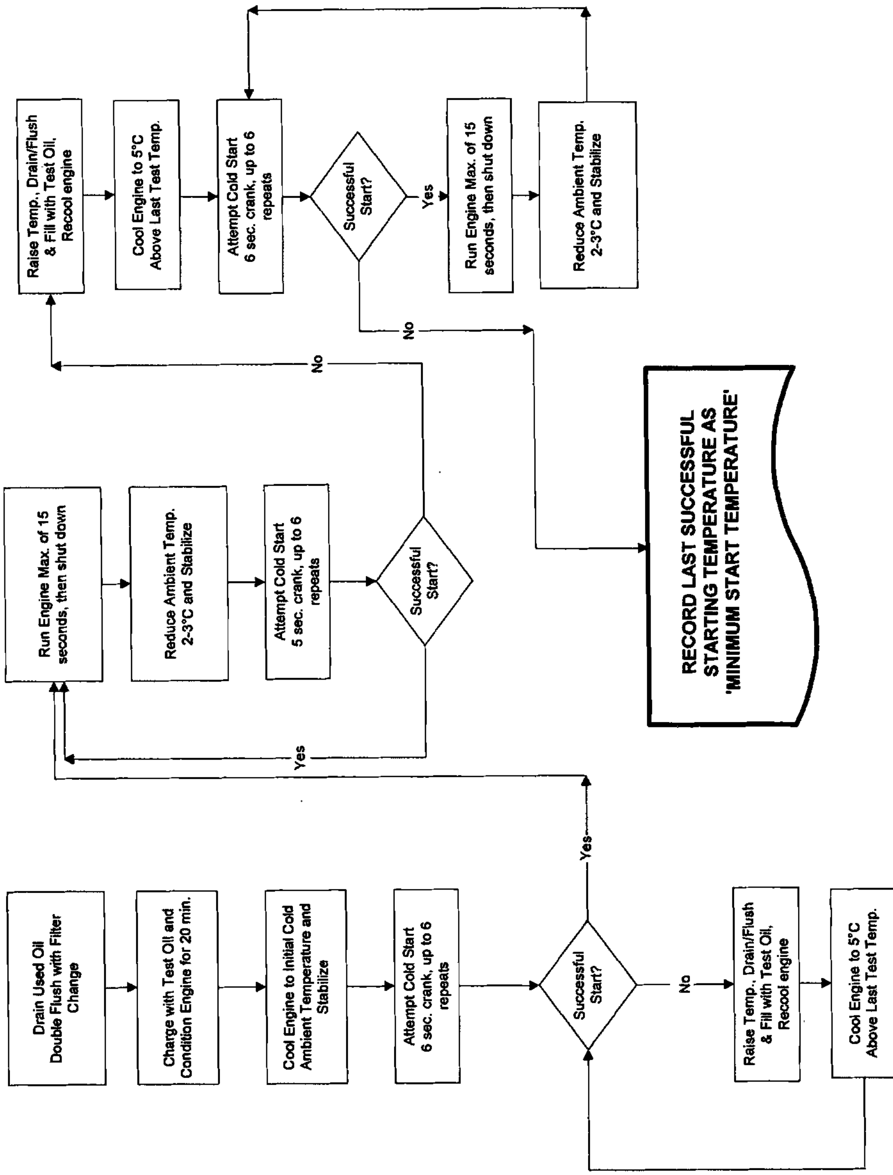


Figure 2 - *Flow Chart for Cold Start Testing Program*

There were two phases to the pumpability studies. Phase I included testing of LTEP 1-7 oils under rapid, overnight cooling profiles, at or below the MSTs determined from the cold start testing. Test temperatures were decreased in 3°C increments from the MST for the oil/engine combination, with repeat testing at one temperature to assess repeatability. Phase II, covered gelation prone oils in response to American Automobile Manufacturers' Association concerns [9], and was accomplished by testing of the LTEP 20 series oils under both rapid and slow cool conditions. These test conditions were somewhat unique to each lab, and are reviewed in detail in the paper covering the Phase II pumpability results [10].

References

[1] *Cold Starting and Pumpability Studies in Modern Engines, ASTM Research Report RR-D02-1442*, November, 1998.

[2] Alexander, D. L., "A Critical Assessment of the Low-Temperature Viscosity Requirements of SAE J300 and Its Future," SAE Paper No. 932794.

[3] *Low Temperature Pumpability Characteristics of Engine Oils in Full Scale Engines, ASTM DS-57*, American Society for Testing and Materials, Philadelphia, PA 19103, September 1975.

[4] Alexander, D. L., "Proposal for an ASTM Investigation of the Low-Temperature Cranking and Pumping Viscosity Requirements of Light-Duty Gasoline and Heavy-Duty Diesel Engines," ASTM D02 Subcommittee 7 meeting June 24, 1991.

[5] Stewart, R. M., "Engine Pumpability and Crankability Tests on Commercial "W" Graded Engine Oils Compared to Bench Test Results," SAE 780369.

[6] Caracciolo, F. and McMillan, M. L., "Effect of Engine Oil Viscosity on Low-Temperature Cranking, Starting and Fuel Economy." SAE Paper No. 790728.

[7] Stover, W. H., "Cold Starting and Oil Pumpability - an Evaluation of New and Used Oils in Gasoline Engines." SAE Paper No. 790729.

[8] May, C. J., Habeeb, J. J. and White, A. M., "Engine Oil Low Temperature Pumpability: A Comparison of SAE 10W30, 5W30 and 0W30 Multigrade Engine Oils," Lubrication Engineering, 43, 7, 557, 1987.

[9] Steiger, J. P., *Letter to R. B. Rhodes, ASTM D02.070C, Engine Oil Panel Selection Chairman*, Sept. 16, 1993.

[10] Rhodes, R B., De Paz, E. F., Girshick, F. W., Henderson, K. O., May, C. J., Tseregounis, S., and Ying, L. H., "Cold Starting and Pumpability Studies in Modern Engines - results from the ASTM D02.07C Low Temperature Engine Performance Task Force Activities: Phase II Pumpability Testing, " Oil Flow Studies at Low Temperatures in Modern Engines, ASTM STP 1388, H. Shaub, Ed., American Society for Testing and Materials, West Conshohocken, PA, 2000.

Fred W. Girshick,[1] Enrique F. De Paz,[2] Christopher J. May,[3] Kenneth O. Henderson,[4] Robert B. Rhodes,[5] Spyros Tseregounis,[6] and Lisa H. Ying[7]

Cold Starting and Pumpability Studies in Modern Engines - Results from the ASTM D02.07C Low Temperature Engine Performance Task Force Activities: Test Oil Selection and Rheological Analysis

---

Reference: Girshick, F. W., De Paz, E. F., May, C. J., Henderson, K. O., Rhodes, R. B., Tseregounis, S., and Ying, L. H., "**Cold Starting and Pumpability Studies in Modern Engines - Results from the ASTM D02.07C Low Temperature Engine Performance Task Force Activities: Test Oil Selection and Rheological Analysis,**" *Oil Flow Studies at Low Temperatures in Modern Engines, ASTM STP 1388*, H. Shaub, Ed., American Society for Testing and Materials, West Conshohocken, PA, 2000.

Abstract: Two series of test oils were obtained for the Low Temperature Engine Performance (LTEP) task force activities: (a) SAE 0W-30, 5W-30, 10W-30, 15W-40, 20W-50 and 25W-30 grades were fully on-specification commercial oils (LTEP 1-7), used in cold starting and Phase I pumpability studies, (b) experimental test oils for Phase II pumpability work (LTEP 20 series) either did not meet the then-current SAE J300 pumping viscosity (TP-1 MRV) limits, or showed significant D5133 gelation index values, and included nominal SAE 5W-30, 10W-30 and 15W-40 grades. LTEP participating labs provided rheological data on the oils including measurements by D 445, D 3829, D 4684, D 5133, and D 5293. Multi-temperature MRV and CCS measurements allowed correlation to the exact engine test temperatures by fitting viscosity and yield stress data to equations. The functional form of the equations is presented and explained, and the fitted coefficients are tabulated. Gelation index and gelation index temperature values, were simply averaged for each oil. For the D 3829 and D 4684 methods that measure yield stress in addition to viscosity, an adjustment was made to the measured viscosity before fitting, to allow for yield stress effects.

---

[1] HD Formulation Science Leader, Infineum USA LP, P.O. Box 536, Linden, NJ 07036.
[2] Project Leader, Consumers Union, 101 Truman Avenue, Yonkers, NY 10703-1057.
[3] Senior Research Associate, Imperial Oil, Research Dept., P.O. Box 3022, Sarnia, Ont., Canada N7T 8C8.
[4] Vice President of Technology, Cannon Instrument Co., P.O. Box 16, State College, PA 16804-0016.
[5] Consultant, Infineum, USA LP, 11735 Normont Dr., Houston, TX 77070.
[6] Staff Research Engineer, General Motors Research Laboratories, P.O. Box 9055, Warren, MI 48090-9055.
[7] Senior Statistician, Infineum USA LP, P.O. Box 536, Linden, NJ 07036.

Introduction

The results of the Low Temperature Engine Performance (LTEP) task force studies were documented in a detailed research report [*1*], which underwent peer review prior to publication. These results are summarized in the following two sections.

Summary, Part I

The following conclusions were made based upon several 1993-94 model year commercial automotive gasoline engines and commercially available engine oils (5 multigrade and 1 monograde) used in the cold start and Phase I pumpability testing of this study. These test oils met the SAE J300 limits current at the time of their solicitation (1993-94):

(1) Of the engines tested, one was found to be significantly different in both starting and pumping characteristics, and was associated with an older engine design.

(2) The modern design engines started, on average, at lower temperatures than earlier engine designs.

(3) Unlike older engines, the minimum starting temperatures of the modern engines were found to be independent of engine characteristics such as number of cylinders, configuration or displacement.

(4) Cold cranking simulator (CCS) viscosity correlated with engine startability. However, the starting viscosities were considerably higher than SAE J300 APR97 limits. In light of these findings, it is recommended that the cold cranking viscosity/temperature limits be revisited.

(5) These oils demonstrated acceptable pumpability at temperatures 5 to 9°C lower than the minimum starting temperatures using two pumpability criteria (150 kPa at pump-out in less than 15 sec. or 10 kPa at near galley in less than 60 sec.). Therefore, for the engines and oils tested at the prescribed conditions, an operational safety margin exists between starting and pumping.

(6) Analysis of the near galley pressurization data indicated that the modern engine designs had limiting pumpability viscosities in the range of 71 to 131 Pa·s (as measured by the Standard Test Method for Predicting the Borderline Pumping Temperature of Engine Oil D 3829) with an average of approximately 93 Pa·s.

(7) The older engine design, which showed higher minimum starting temperatures, had limiting pumpability viscosities in the range of 31 to 48 Pa·s with an average of approx. 43 Pa·s. This limiting viscosity is more consistent with earlier pumpability limits established in ASTM D-57 (2).

Summary, Part II

The following conclusions were made based upon limited testing of 5 multigrade oils in 7 commercial automotive gasoline engines used in the Phase II pumpability testing. These experimental test oils were selected because they demonstrated structure in certain bench tests such as TP-1 1 MiniRotary Viscometer (MRV) and/or Scanning Brookfield procedures:

(1) Under appropriate cooling conditions and operating conditions, air-binding pumpability could be generated in certain engine types.
(2) Air-binding conditions are strongly affected by oil height above the pick-up tube; very low oil charge exacerbates air-binding tendencies.
(3) Air-binding was only observed when significant structure was detected in the oils (>40 gel index, MRV Yield stress 70-105 Pa).

Impact of LTEP Findings

While the formal LTEP findings were published only in November, 1998, meetings throughout the lifetime of the task force served as a focal point for review and discussion of the results as they were produced. Thus, in mid-1995, when it became apparent that, on average, the modern engine designs were starting at lower temperatures thus affecting the pumpability safety margin, SAE acted on this information, and after considering both cold start and pumpability data, successfully balloted modifications to the MRV "W" grade measuring temperatures and viscosity limits [*2*]. More recently, SAE has been considering modifications to CCS "W" grade measurement temperature and viscosity limits, again to reflect cold starting data from the LTEP task force.

It should also be noted that the LTEP task force has shared information, including test protocols, with European groups considering similar studies on cold starting and pumpability [*3*].

Acknowledgments

The successful completion of this complex and costly enterprise would not have been possible without the active participation and support of the individual LTEP task force members. Special thanks are given to the working group members who laboured tirelessly over the LTEP research report, and to all those who provided comments and corrections, which added to the value of the report.

References

[1] *Cold Starting and Pumpability Studies in Modern Engines, ASTM Research Report RR-D02-1442*, November, 1998.
[2] *SAE J300, Engine Oil Viscosity Classification, Rev. Dec95.*
[3] May, C. J., Minutes of ASTM Committee D-2, Subcommittee 7, Section C, Low Temperature Engine Performance (LTEP) Task Force, December 9, 1996.

Spyros Tseregounis,[1] Enrique F. De Paz,[2] Fred W. Girshick,[3] Kenneth O. Henderson,[4] Christopher J. May,[5] Robert B. Rhodes,[6] and Lisa H. Ying[7]

Cold Starting and Pumpability Studies in Modern Engines - Results from the ASTM D02.07C Low Temperature Engine Performance Task Force Activities: Cold Start Testing

---

Reference: Tseregounis, S., De Paz, E. F., Girshick, F. W., Henderson, K. O., May, C. J., Rhodes, R. B., and Ying, L. H., **"Cold Starting and Pumpability Studies in Modern Engines - Results from the ASTM D02.07C Low Temperature Engine Performance Task Force Activities: Cold Start Testing,"** *Oil Flow Studies at Low Temperatures in Modern Engines, ASTM STP 1388,* H. Shaub, Ed., American Society for Testing and Materials, West Conshohocken, PA, 2000.

Abstract: Cold start studies were conducted on 10 different engines (including duplicates of one type), and six different multigrade oils ranging from SAE 0W-30 to 25W-30. As expected, the results showed that for all the engines, the minimum starting temperature decreased with decreasing winter viscosity grade. The 4.0L I-6 engine had the highest minimum start temperatures (MSTs) for all the oils, while the lowest MSTs were distributed over seven of the other eight engines tested. The difference in the starting characteristics of the 4.0L I-6 engine were attributed to the fact that this engine design was relatively old (early 1980s) compared to the rest of the engines in this program (1990s). Another interesting observation was that, excluding the 4.0L I-6 engine, there was no effect on the MSTs due to engine displacement, number of cylinders or configuration. This is contrary to older studies which showed that as number of cylinders increased, minimum starting temperatures decreased. Improved starting characteristics of these modern engines are attributed to improvements in engine technology.

[1] Staff Research Engineer, GM Research & Development and Planning, P.O. Box 9055, Warren, MI 48090-9055.

[2] Project Engineer, Consumers Union, 101 Truman Avenue, Yonkers, NY 10703-1057.

[3] HD Formulation Science Leader, Infineum USA LP, P.O. Box 536, Linden, NJ 07036.

[4] Vice President of Technology, Cannon Instrument Co., P.O. Box 16, State College, PA 16804-0016.

[5] Senior Research Associate, Imperial Oil, Research Dept., P.O. Box 3022, Sarnia, Ont., Canada N7T 8C8.

[6] Consultant, Infineum, USA LP, 11735 Normont Dr., Houston, TX 77070.

[7] Senior Statistician, Infineum USA LP, P.O. Box 536, Linden, NJ 07036.

An excellent correlation was observed between minimum start temperature and cold cranking simulator (CCS) viscosity for the modern engines. In addition, the CCS viscosity for these engines at the minimum start temperatures was 2 to 4 times higher than the present SAE J300 limits. In contrast, the MSTs for the older engine design (4.0L I-6) appear to follow the present CCS temperature and viscosity limits. The oil viscosity at the MST for the 4.0L I-6 engine was relatively constant (3,000 to 5,000 mPa-s) throughout the whole temperature range, while with the newer engines, limiting viscosity decreased with decreasing temperature (25,000 mPa-s at -19°C to 7,000 mPa-s at -37°C). This requirement for lower oil viscosities at lower temperatures is attributed to a need to compensate for increased internal engine friction with decreasing temperature.

Comparison of these results with those from the 1970s shows that the older engine design (4.0L I-6) follows a similar pattern (comparable CCS viscosities and no temperature effect) to the old 4- and 6-cylinder engines, while the newer engines parallel the results for older V-8s.

Keywords: starting, engine startability, low temperature, CCS viscosity, cold cranking, engine oil, gasoline engine

Background

As discussed in the introductory paper in this series [*11*], a 1992 request to ASTM by the Society of Automotive Engineers' Fuels and Lubricants Division asked that Subcommittee D02.07 and its appropriate task forces organize and conduct a low-temperature engine test program [*1, 2*]. This request was motivated by independent studies published in the early 1990's which suggested that the safety margin between cold starting (cranking) and pumpability built into the SAE J-300 specification might not be valid for modern engines [*3-5*]. In his May 1992 letter [*1*], Sheahan cited five changes in engines and lubricants which could be lowering the starting temperatures of these engines including low-friction engine designs, fuel injection systems, computer control of fuel flow and ignition, high power-density starting motors and batteries, and friction-modified engine oils. SAE specifically asked ASTM to address four goals, the key one for this paper being "to determine if modern engines start (on average) at lower temperatures than have earlier engine designs" [*1*].

To address SAE's requests, the Low Temperature Engine Performance task force was established within Section D02.07C in June 1992, and actively worked on these issues through 1998 [*2*]. Cold start testing was conducted under the direction of the Light Duty Engine Testing Panel within the LTEP task force. Work conducted under the auspices of this task force was funded by individual participants.

In this paper we report the results of these cold starting studies of modern engines with current technology engine oils.

Experimental Details

Oil Selection

The selection and procurement of test oils was the responsibility of the LTEP Oil Selection Panel, and these activities are discussed in detail in an earlier paper in this series [*12*] (see also [*2*]). For engine starting studies it was agreed that a wide selection of multigrade oils should be represented in the test program in order to meet primary task force objectives, and the panel requested a range of fully on-specification commercial oils. In order to ensure that the evaluation not be limited to a single type of additive or basestock, donations were restricted to one viscosity grade per company. The 6 multigrade oils obtained for this study along with the assigned LTEP oil codes were as follows:

SAE Grade	0W-30	5W-30	10W-30	15W-40	20W-50	25W-30
LTEP #	1	2	3	5	6	7

It should be noted that LTEP 7 is a straight grade SAE 30 engine oil which also meets the low temperature requirements of an SAE 25W oil, and was included to specifically address SAE's concerns regarding starting and pumpability of monogrades.

Test Oil Rheology

Rheological properties of all the LTEP test oils are discussed in another paper in this series [*12*] and in the research report [*2*]. CCS measurements at multiple temperatures were obtained for the test oils. The rheological data for each oil (viscosity-temperature) were fitted to a modified MacCoull-Walther-Wright equation that allows curvature with the data centered to minimize computer rounding errors [*2, 12*]. This allowed interpolation to predict CCS viscosity at the exact temperatures at which the engines started. In general, the viscosities were measured at integral temperatures, most often multiples of 5°C, while the temperatures of the engine tests were usually recorded at multiples of 0.1°C. Mean CCS viscosity values for each LTEP oil at 5°C intervals are shown in Table 1, while Table 2 lists the correlation constants and equations as derived from the raw data [*2,12*].

Table 1 - *CCS (D 5392) Viscosities, mPa-s (Mean Values)*

Temperature, °C	LTEP 1 0W-30	LTEP 2 5W-30	LTEP 3 10W-30	LTEP 5 15W-40	LTEP 6 20W-50	LTEP 7 25W-30
-40	9,700	---	---	---	---	---
-35	4,900	15,000	---	---	---	---
-30	2,700	6,800	15,100	---	---	---
-25	1,600	3,200	6,500	14,200	---	---
-20	---	1,600	3,100	6,600	13,500	28,500
-15	---	---	1,700	3,300	6,900	12,500
-10	---	---	---	1,700	3,800	6,000
-5	---	---	---	---	2,100	3,200

Table 2 - *CCS (D 5392) Viscosity-Temperature Correlation Constants*

	LTEP 1	LTEP 2	LTEP 3	LTEP 5	LTEP 6	LTEP 7
	0W-30	5W-30	10W-30	15W-40	20W-50	25W-30
Intercept, a	0.4799	0.5055	0.5438	0.5822	0.6204	0.6489
Linear, b	-3.0779	-4.7076	-4.2192	-4.1917	-4.2750	-4.3460
Quadratic, c	8.3218	-13.7903	10.6377	-5.0085	14.6062	5.9449
N included	9	12	15	12	12	22
N dropped	0	0	0	0	1	0
Min-Max	-40 -25	-35 -20	-30 -15	-25 -10	-20 -5	-24 -5
R-squared	0.9968	0.9986	0.9986	0.9989	0.9988	0.9983
Root MSRE	0.001768	0.001440	0.001390	0.001161	0.001158	0.001914
W = a + b*T + c*T^2 where W = Log[Log(mPa-s)] and T = Log[(273.15+Celsius)/(273.15+Center)] Center = -20 for CCS						

Engine Selection

Details on the selection of engines for the startability work are given elsewhere [*2, 13*]. The engines, selected from a list of recommended engines, are shown in Figure 1 and Table 3. It should be noted that the selections encompass 1993/1994 model year 4-, 6-, and 8- cylinder designs. All engines were fuel injected systems. Oil flow schematics for the test engines are included in the ASTM report [*2*].

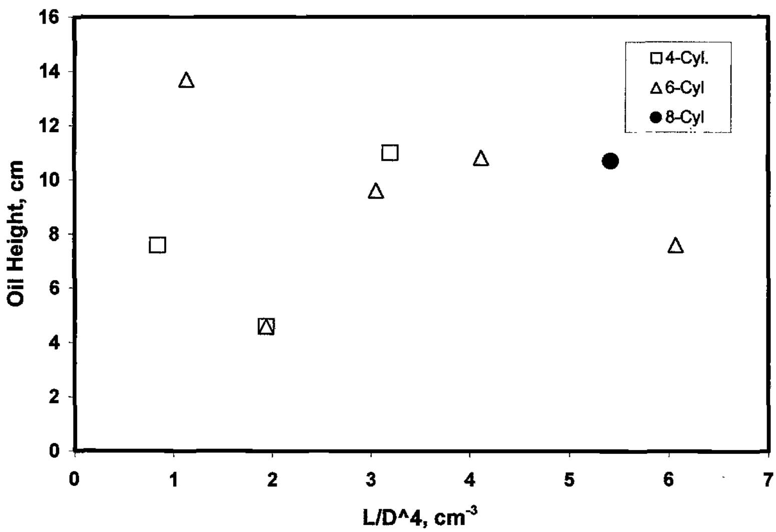


Figure 1 - *Oil Pick-up Tube Length/(Diameter^4) versus Oil Height for Selected Engines*

Table 3 - *Test Lab - Engine Combinations for Cold Start Testing*

Test Lab	Facility	Engine Type	Manufacturer	Model Year
Imperial Oil /Exxon	Cold Room	1) 3.3L V6 OHV 2) 2.3L I4 OHC 3) 3.8L V6 OHV 4) 1.9L I4 OHV 5) 4.6L V8 OHC 6) 3.0L V6 OHC	1) D 2) F 3) F 4) E 5) E 6) H	1) 1994 2) 1993 3) 1994 4) 1994 5) 1994 6) 1993
Shell Westhollow Tech Center	Cold Room	1) 4.0L I6 OHV 2) 3.8L V6 OHV	1) D 2) F	1) 1994 2) 1994
Texaco	Cold Box	1) 2.2L I4 OHC	1) H	1) 1994
Southwest Research Inst.	Cold Box	1) 3.0L V6 OHV	1) E	1) 1993

Test Protocols

Standardized protocols for conducting cold start testing were developed which included instrumentation/sampling, engine and oil conditioning, cooling rates, starting/running procedures, and fuel selection. (see [*2, 13*] for details). In order to meet the requirements laid out by SAE/ASTM, fired cold start studies were conducted to establish minimum starting temperatures (MST) for each vehicle/oil combination. A breakdown of the participating laboratories and vehicles tested is included in Table 3.

Care was taken to closely control operating variables including engine state-of-tune, fuel characteristics, and battery condition to ensure adequate test precision in a "real-world" atmosphere: (i) engines were given a complete tune-up prior to testing, with special attention to the fuel delivery system and throttle plate assembly, (ii) fuel distillation characteristics were selected to ensure adequate volatility, typical of current commercial practice in winter conditions in the northern U.S. and Canada[8] , (iii) the use of warm booster batteries removed a significant source of potential test variation, but also resulted in a more severe test [*6-9*]. Because two different labs were able to test a 3.8L V-6 from manufacturer F, engine-to-engine variation could be assessed. Including these duplicates, 10 engines were used in the cold starting tests at four different labs.

Engine instrumentation allowed rpm, oil temperature and pressure to be monitored at five times per second during the test. The testing protocol called for conducting up to six 5-second cranking attempts for each vehicle/oil combination at a particular temperature. If cold start was achieved, the engine was shut down, the temperature lowered by 2-3°C and stabilized, then a new starting attempt was made. Once a no-start condition had been reached, the vehicle underwent a complete fresh oil/filter change and received a new set of spark plugs, followed by repeat cold start evaluation at/below the MST achieved in the

[8] Fuel was supplied by Shell Development (for Shell testing), and Imperial Oil (for testing by Imperial Oil, Southwest Research Institute, and Texaco). Fuel characteristics are compared in Table 4.

first test. A minimum of three complete cold starting tests on each oil/vehicle combination allowed average MSTs to be determined with greater accuracy.

Table 4 - *Fuel Characteristics - Cold Start Testing (as measured by donating lab)*

Property	*Imperial Oil*	*Shell Westhollow*
T_{10}, °C	40	42.2
T5, °C	98	96.1
RVP, psi	---	10.79

Results and Analysis

The main purpose of this part of the program was to determine the minimum start temperature (MST) for each engine with oils LTEP 1 through LTEP 7. The MSTs were then correlated to the Cold Crank Viscosities of the oils, using the rheological data of the oils.

Except for temperature, all other engine conditions during these experiments were set to maximize the potential for having the engines start. Good winter service fuel was used for all testing. The spark plugs were replaced and/or refurbished before each test. All parts of the air intake system were cleaned just before each test to remove any frost which may have formed during the cool down cycle, and a fully charged and warm (room temperature) battery was used to drive the starter motor. Because of these ideal conditions, the MSTs measured in this program can be expected to be lower than those encountered in the normal real world operation of these engines. Thus, any conclusions or correlations generated from this program represent the most extreme cases that may be encountered in the field.

Generally, the procedure involved rapid cooling of the engine or vehicle to the test temperature and then holding isothermal conditions for 12 to 16 hours (Figure 2 shows a typical oil cooling profile for startability testing). A start attempt was then made.

This process was repeated at lower and lower temperatures until the engine would not start after six consecutive cranking attempts. Figure 3 shows typical cold cranking engine data (RPM vs. Time) at three different temperatures. These results demonstrate the transition from start to no start conditions. At -19°C, the engine starts with the very first crank attempt. The engine is then allowed to run for an additional few seconds and shut down. In the next experiment, when the temperature is reduced to -20°C, two cranking attempts are necessary to start the engine.

Again, the engine is allowed to operate for a short while before being turned off. Finally, at -21°C the engine does not start, even after six cranking attempts. Thus, the MST for this oil/engine combination is -20°C.

During the initial work in this program there was some concern that, in cases where multiple cranking attempts were used to start the engine, the fuel injected during the failed ignition attempts passed around the rings and into the oil reducing its viscosity. This could increase the potential for the engine to start with subsequent cranking attempts. Since fuel dilution would be the highest in the cases where the engine did not

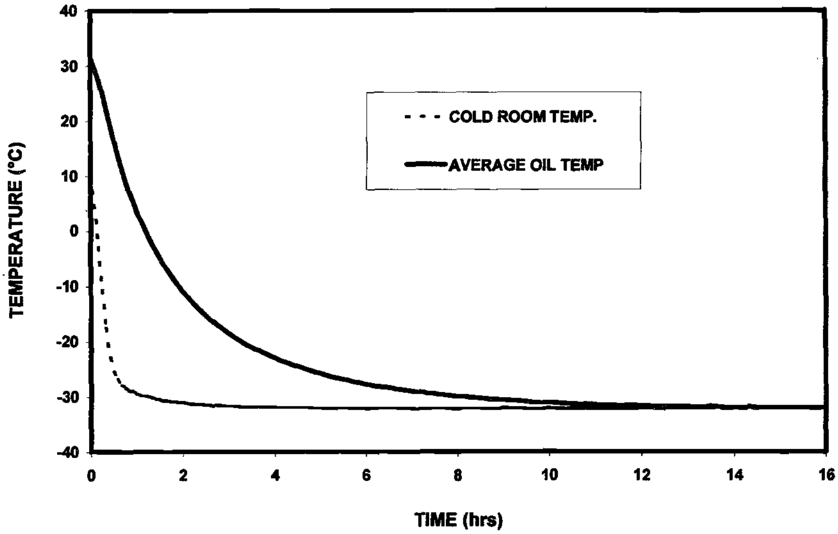


Figure 2 - *Typical Cooling Profile for Startability Testing*

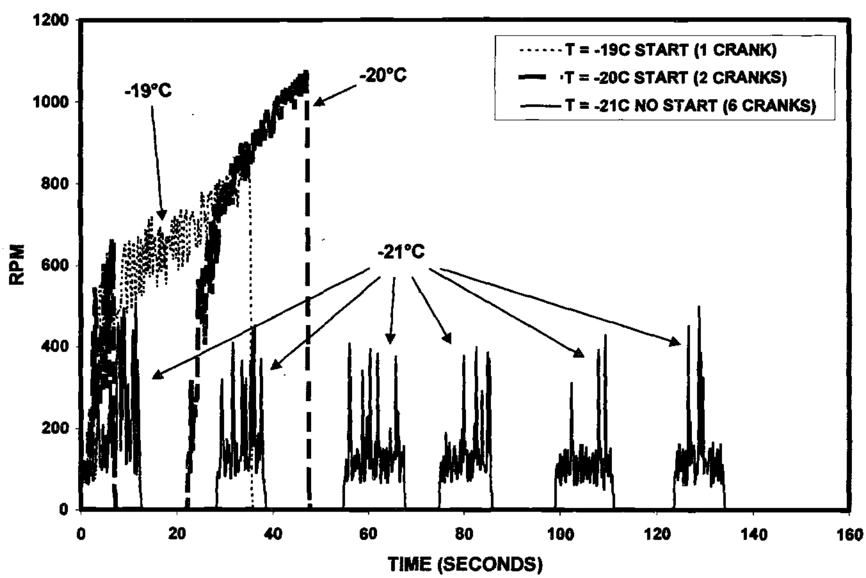


Figure 3 - *Typical Startability Testing Results as Temperature Lowered (2.2L I4 Engine, LTEP 6)*

start after six cranking attempts, some of these oil drains were analyzed for fuel content [2]. On average, the results showed fuel dilution to be less than 2 wt.%.

To obtain some measure of the repeatability of the MSTs, most oil/engine combinations were tested three times. Table 5 demonstrates the typical results for the 1.9L I-4 engine.

Table 5 - *Typical Minimum Start Results (Engine: 1.9L I-4)*

Oil ID and SAE Grade		Minimum Start Temperatures (°C)				
		Trial 1	Trial 2	Trial 3	*Average*	*Std. Dev.*
LTEP 1	0W-30	<-39.1			*-39.1*	
LTEP 2	5W-30	-33.1	-31.7	-33.2	*-32.7*	*0.84*
LTEP 3	10W-30	-23.5	-27.2	-28.7	*-26.5*	*2.68*
LTEP 5	15W-40	-26.9	-26.6	-24	*-25.8*	*1.59*
LTEP 6	20W-50	-23.1	-21.8	-21.5	*-22.1*	*0.85*
LTEP 7	25W-30	-20.2	-19.7	-19.6	*-19.8*	*0.32*

For detailed results of all the engines, the reader is referred to the ASTM Research Report, Appendix B [2]. Note the "less than" (<) sign in front of trials with oil LTEP 1. This is an indication that the engine started at the lowest attainable oil temperature (the temperature shown after the sign) for the cold room at the particular laboratory.

The average MSTs for each oil/engine combination are presented in Table 6 and plotted in Figure 4. An overall MST and standard deviation for each oil is also included at the bottom of Table 6.

Table 6 - *Average Minimum Starting Temperatures (°C)*

Engine ID	LTEP 1	LTEP 2	LTEP 3	LTEP 5	LTEP 6	LTEP 7
	0W-30	5W-30	10W-30	15W-40	20W-50	25W-30
1.9L I-4E	<-39.1	**-32.7**	-26.5	-25.8	-22.1	-19.8
2.2L I-4G	-37.0	-31.7	-26.0	-25.0	-19.7	-18.3
2.3L I-4F	<-37.8	-31.7	-26.3	-25.7	**-22.9**	-20.8
3.0L V-6H	-37.2	-31.6	-25.3	-24.0	-21.7	-20.5
3.0L V-6E	**	**	**-30.7**	-23.5	-20.1	-17.5
3.3L V-6I	<-36.2	-30.3	-27.3	-25.5	-20.8	-19.9
4.6L V-8E	**<-39.2**	-31.1	-27.7	-25.2	-22.6	**-22.3**
*3.8L V-6F	-32.6	-29.4	**-30.7**	-23.7	-22.4	-17.8
*3.8L V-6F	<-38.5	-32.1	-28.9	**-26.5**	**-22.9**	-18.8
4.0L I-6I	**-30.2**	**-28.5**	**-23.1**	**-18.0**	**-11.9**	**-6.7**
Overall Avg.	*-36.4*	*-31.0*	*-27.3*	*-24.3*	*-20.7*	*-18.2*
Overall Std Dev.	*3.1*	*1.4*	*2.4*	*2.4*	*3.3*	*4.3*

* Same engine model tested in two different laboratories.

** Laboratory did not test these oils

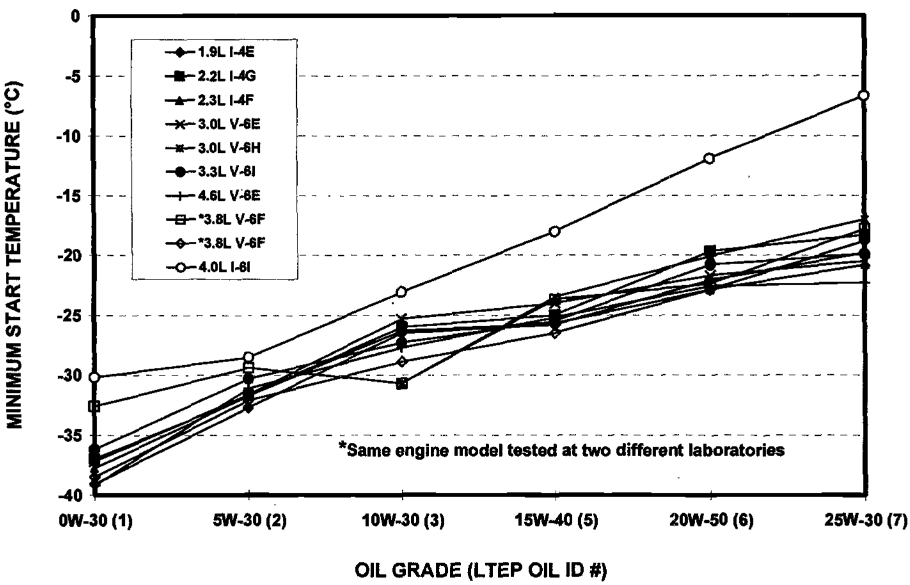


Figure 4 - *Minimum Starting Temperatures for LTEP Oils and Engines*

In cases where an absolute minimum starting temperature could not be determined (< sign), the minimum attainable temperature was used as the MST to calculate the overall average and standard deviation.

Before initiating any discussion of the results, some comments on the format used in the table are appropriate. The numbers in **bold** indicate the engines which had the lowest (coldest) starting temperature with the particular oil, while those shown in **bold with a frame around them**, indicate the engines which had the highest **(warmest)** starting temperature for the particular oil. Duplicate results are shown for the 3.8L V-6 engine because two of these engines were tested (one each in two labs).

As expected, the results show that for all the engines, the minimum starting temperature decreases with decreasing winter viscosity grade. The 4.0L I-6 engine has the highest MSTs for all the oils, while the lowest MSTs are distributed over seven of the other eight engines tested. This is clearly evident in Figure 4, where it appears that the 4.0L I-6 engine lies outside the rest of the engine population. In fact, evaluation of the results using statistical t-tests revealed that the 4.0L I-6 MSTs are significantly different from the rest of the engines.

These same statistics also confirmed that the MSTs for the rest of the engines were not significantly different from each other and could be averaged into a single MST for all the engines with each oil. Table 7 shows how the MSTs and their average and standard deviation will change if the 4.0L I-6 engine is excluded.

Table 7 - *Average Minimum Starting Temperatures (°C), Excluding 4.0L I-6 Engine*

Engine ID	LTEP 1	LTEP 2	LTEP 3	LTEP 5	LTEP 6	LTEP 7
	0W-30	5W-30	10W-30	15W-40	20W-50	25W-30
1.9L I-4E	<-39.1	**-32.7**	-26.5	-25.8	-22.1	-19.8
2.2L I-4G	-37.0	-31.7	-26.0	-25.0	**-19.7**	-18.3
2.3L I-4F	<-37.8	-31.7	-26.3	-25.7	**-22.9**	-20.8
3.0L V-6H	-37.2	-31.6	**-25.3**	-24.0	-21.7	-20.5
3.0L V-6E	**	**	**-30.7**	**-23.5**	-20.1	**-17.5**
3.3L V-6I	<-36.2	-30.3	-27.3	-25.5	-20.8	-19.9
4.6L V-8E	**<-39.2**	-31.1	-27.7	-25.2	-22.6	**-22.3**
*3.8L V-6F	**-32.6**	**-29.4**	**-30.7**	-23.7	-22.4	-17.8
*3.8L V-6F	<-38.5	-32.1	-28.9	**-26.5**	**-22.9**	-18.8
Overall Avg.	*-37.2*	*-31.3*	*-27.7*	*-25.0*	*-21.7*	*-19.5*
Overall Std Dev.	*2.2*	*1.1*	*2.0*	*1.0*	*1.2*	*1.7*

* Same engine model tested at two different laboratories.
** Laboratory did not test these oils

Note that without the 4.0L I-6 engine the highest starting temperatures for each oil are now just about evenly distributed among all the engines. The small overall standard deviation (1 to 2°C) at the bottom of this table again demonstrates the insignificant differences in MSTs between these engines. The difference in the starting characteristics of the 4.0L I-6 engine may be attributed to the fact that this engine design is relatively old (early 1980s) compared to the rest of the engines in this program (1990s).[9]

The apparent difference in the MSTs (especially at the lower viscosity grades) for the two 3.8L V-6 engines were also determined not to be significantly different from each other or from the rest of the engine population (excluding the 4.0L I-6 engine). At the time when those experiments were being performed, there was some concern over these differences. It was discovered that while both laboratories were using winter grade fuel, there were slight volatility differences between the two fuels (Table 8).

Table 8 - *Fuel Volatility Characteristics*

Laboratory	Reid Vapor Pressure	Temperature Cut-Points (°C)	
		10% Vaporized	50% Vaporized
Imperial Oil	13.97	37	84
Texaco FLTD	"	"	"
Southwest Research Institute	"	"	"
Shell Westhollow Tech Center	10.79	42	96

[9] Private communication from engine manufacturer.

The laboratory using the fuel having the higher vapor pressure and lower cut temperatures (i.e., higher volatility) had the lower MSTs. After discussing these differences with fuel experts in several locations, it was concluded that these differences in fuel volatility could be the reason for the discrepancy in the MSTs for these engines. Because of this conclusion and the subsequent determination that the MSTs were not statistically different, no other attempts were made to test any of these engines with the fuel from the other laboratory.

Another interesting observation from all these results is the fact that (again excluding the 4.0L I-6 engine) there appears to be no effect on the MSTs due to engine displacement (Figure 5), number of cylinders or configuration (Figure 6). This is contrary to previous studies which showed that the more cylinders an engine had, the lower its MST, i.e., eight cylinder engines had lower MSTs than six or four cylinder engines [*8,9*]. The improved starting characteristics of these modern engines are attributed to improvements in engine technology, particularly improved starter motors, fuel injection and electronic controls.

Figure 7 shows the minimum start temperatures for the 4.0L I-6 engine, the average MSTs for the other eight engines and the temperatures used to measure the Cold Crank Simulator (CCS) and the Mini-Rotary Viscometer (MRV) viscosities for the various oil grades. One set of MRV temperatures (SAE J300 December '94) correspond to those in use at the time this program was initiated, and the other (SAE J300 December '95) are

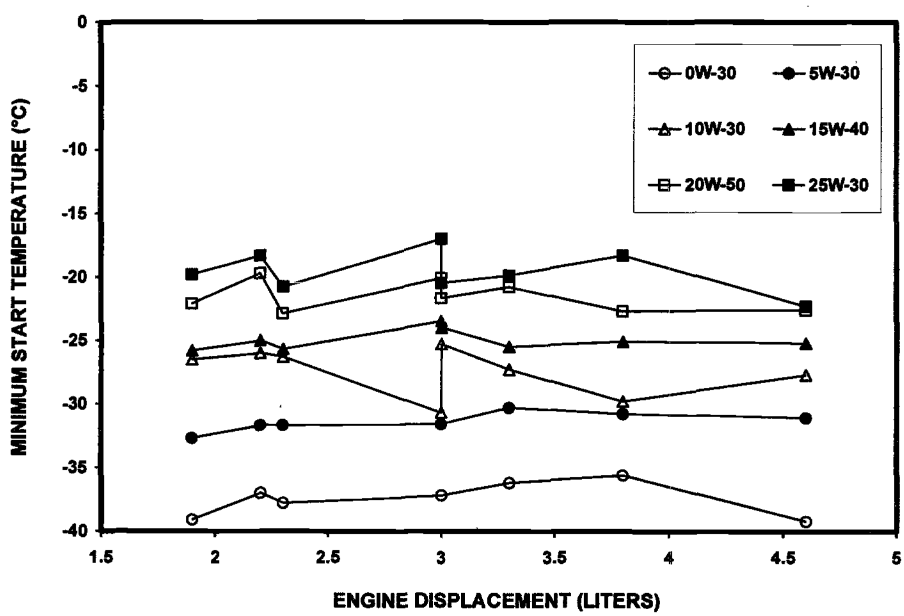


Figure 5 - *Correlation Between Engine Displacement and Minimum Start Temperature (Excluding 4.0L I6 Engine I)*

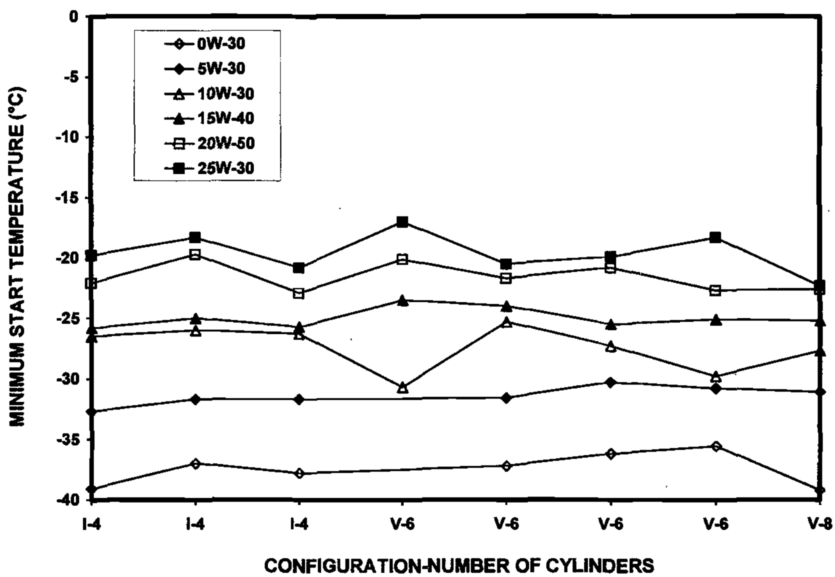


Figure 6 - *Correlation Between Configuration-Number of Cylinders and Minimum Start Temperature (Excluding 4.0L I6 Engine I)*

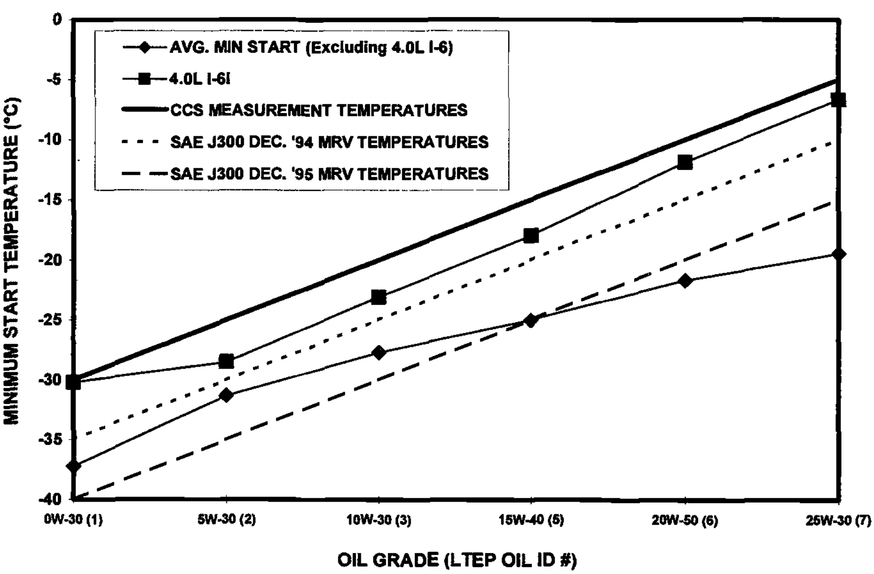


Figure 7 - *Average Minimum Start Temperatures for LTEP Oils and Engines*

those implemented in response to the startability results from this program (see the ASTM report for details [2]). Remembering that all the conditions used during this testing were set to maximize the potential for engine starting, it appears that the temperatures shown on the graph for measuring the CCS and the old MRV viscosities would adequately protect the 4.0L I-6 engine. However, these temperatures appear to be too mild for the rest of the engines. At the time these results were obtained, this was particularly worrisome because all the MSTs were below the MRV measurement temperatures, implying that the engines may start in cold weather but could encounter oil pumping problems. The pumpability portion of the LTEP program was designed to make this determination. As a temporary measure until the pumpability program was completed, all the MRV measurement temperatures were reduced by 5°C (SAE J300 December '95). This temperature reduction placed all the MRV temperatures at or below the measured MSTs for the normal cold weather oil grades (0W to 15W).

The other aspect of this portion of the program that needed to be evaluated was whether or not the Cold Crank Viscometer and/or its present limits were still relevant for newer engines. Figure 8 shows the correlation between the log of CCS viscosity and MSTs. For comparison, the present CCS viscosity and temperature limits are also included, along with the MST vs. CCS viscosity data of similar studies from the 1970s [8, 9]. The figure shows an excellent correlation ($R^2 = 0.98$) between the minimum start temperature and CCS viscosity for modern engines. It also shows that the CCS viscosity for these engines at the minimum start temperatures is 2 to 4 times higher than the present limits.

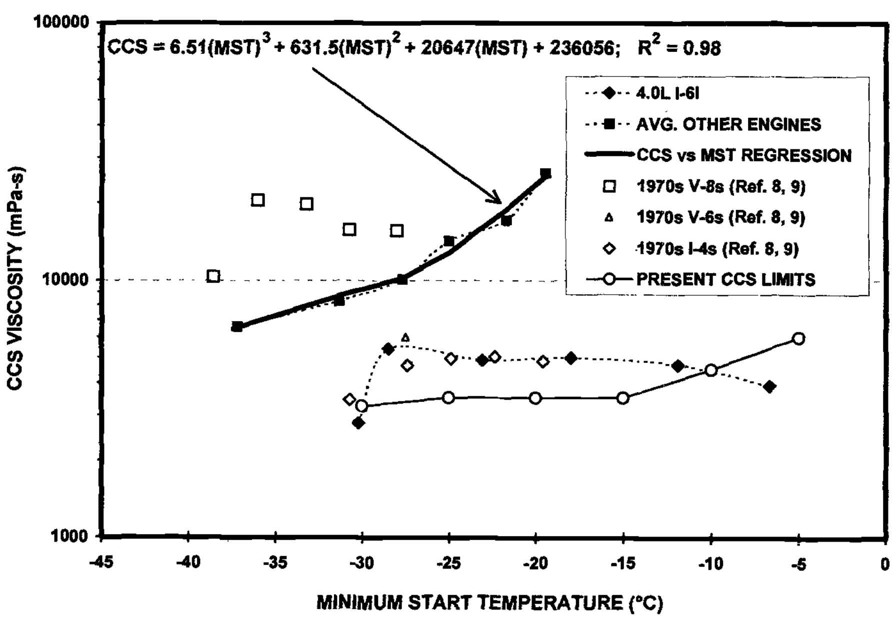


Figure 8 - *Correlation Between Minimum Start Temperatures and CCS*

In contrast, the MSTs for the older engine (4.0L I-6) appear to follow the present CCS temperature and viscosity limits. The other interesting observation in these results is that the CCS viscosity for the 4.0L I-6 engine is relatively constant (3,000 to 5000 mPa-s) throughout the whole temperature range, but that of the newer engines decreases significantly (25,000 mPa-s at -19°C to 7000 mPa-s at -37°C) with decreasing temperature. This requirement for lower oil viscosities at lower temperatures has been attributed to a need to compensate for increased internal engine friction with decreasing temperature.[10] In older engines, it has been shown that decreasing fuel volatility at lower temperatures can contribute to the need for lower viscosity [*9, 10*].

Comparison of these results with those from the 1970s shows that the older engine design (4.0L I-6) follows a similar pattern (comparable CCS viscosities and no temperature effect) to the old four cylinder and six cylinder engines, while the newer engines parallel the results for the older V-8 engine. This again demonstrates how the improvements in the starting systems of modern engines has made all engines (no matter what displacement, number of cylinders, or configuration) as easy to start as the old V-8 engines. In the past, the ease of starting for a V-8 engine was attributed to the increased number of firings per crankshaft revolution.

Conclusions

The following conclusions can be made based upon several 1993-94 model year commercial automotive gasoline engines and commercially available engine oils (5 multigrade and 1 monograde) used in the cold start testing in this study [*2*]. These test oils met the SAE J300 limits current at the time of their solicitation (1993-94):

(1) Of the engines tested, one was found to be significantly different in starting characteristics, and was associated with an older engine design.
(2) The modern design engines started, on average, at lower temperatures than earlier engine designs.
(3) Unlike older engines, the minimum starting temperatures of the modern engines were found to be independent of engine characteristics such as number of cylinders, configuration or displacement.
(4) CCS viscosity correlated with engine startability. However, the starting viscosities were considerably higher than SAE J300 APR97 limits. In light of these findings, it is recommended that the cold cranking viscosity/temperature limits be revisited.

Acknowledgments

Vehicle testing at Imperial Oils Research Labs was supported by funding from Imperial Oil, Exxon Research & Engineering, Paramins and Castrol Inc. Imperial Oil gratefully acknowledges the loan of two vehicles by General Motors Research and Chrysler Engineering for loan of one vehicle in the cold start testing program.

[10] Private communication from engine manufacturer.

Engine/vehicle testing at Shell Westhollow Technology Center was supported by funding from Shell Chemical Co. Engine/vehicle testing at Texaco R&D was supported by funding from Texaco Inc. Cold start engine testing at Southwest Research Institute was internally funded.

References

[1] Sheahan, T. J., Letter to Z. M. Holubec (Chairman, ASTM Subcommittee D02.07) and F.R. Duffey (Chairman, ASTM Subcommittee D02.0B), May 29, 1992.

[2] *Cold Starting and Pumpability Studies in Modern Engines, ASTM Research Report RR-D02-1442*, American Society for Testing and Materials, West Conshohocken, PA, November 1998.

[3] Alexander, D. L., "Proposal for an ASTM Investigation of the Low-Temperature Cranking and Pumping Viscosity Requirements of Light-Duty Gasoline and Heavy-Duty Diesel Engines," ASTM D02 Subcommittee 7 meeting June 24, 1991.

[4] Spearot, J. A., "Engine Oil Viscosity Classification Low-Temperature Requirements - Current Status and Future Needs," *Low Temperature Lubricant Rheology Measurement and Relevance to Engine Operation, ASTM STP 1143*, R.B. Rhodes, Ed., American Society for Testing and Materials, West Conshohoken, PA, 1992, pp. 161-181.

[5] Alexander, A. G., MacAlpine, G. A., May, C. J. and Smith, C. R., "Passenger Car and Heavy Truck Cold Cranking Studies - Effects of Oil Viscosity and Vehicle Type," *Low Temperature Lubricant Rheology Measurement and Relevance to Engine Operation, ASTM STP 1143*, R.B. Rhodes, Ed., American Society for Testing and Materials, West Conshohoken, PA, 1992, pp. 137-160.

[6] Stewart, R. M., "Engine Pumpability and Crankability Tests on Commercial "W" Graded Engine Oils Compared to Bench Test Results," SAE 780369.

[7] Eberan-Eberhorst, C. G. A., Neveu, C., Rossi, A. and Thompson, M., "Low Temperature Starting and Pumpability Requirements of European Automotive Engines," SAE Paper No. 790727.

[8] Caracciolo, F. and McMillan, M. L., "Effect of Engine Oil Viscosity on Low-Temperature Cranking, Starting and Fuel Economy." SAE Paper No. 790728.

[9] Stover, W. H., "Cold Starting and Oil Pumpability - an Evaluation of New and Used Oils in Gasoline Engines," SAE Paper No. 790729.

[10] Stewart, F. R. M. and Meyer, W. A. P., "Effect of Oil Viscosity, Fuel Vapor Pressure and Other Variables on Starting of Four Compact Cars at -10F," SAE Paper No. 660542.

[11] De Paz, E. F., Girshick, F. W., Henderson, K. O., May, C. J., Rhodes, R B., Tseregounis, S., and Ying, L. H., "Cold Starting and Pumpability Studies in Modern Engines - results from the ASTM D02.07C Low Temperature Engine Performance Task Force Activities: Background and Organization," Oil Flow Studies at Low Temperatures in Modern Engines, ASTM STP 1388, H. Shaub, Ed., American Society for Testing and Materials, West Conshohocken, PA, 2000.

[12] De Paz, E. F., Girshick, F. W., Henderson, K. O., May, C. J., Rhodes, R B., Tseregounis, S., and Ying, L. H., "Cold Starting and Pumpability Studies in Modern Engines - results from the ASTM D02.07C Low Temperature Engine Performance Task Force Activities: Oil Selection and Rheology," Oil Flow Studies at Low Temperatures in Modern Engines, ASTM STP 1388, H. Shaub, Ed., American Society for Testing and Materials, West Conshohocken, PA, 2000.

[13] De Paz, E. F., Girshick, F. W., Henderson, K. O., May, C. J., Rhodes, R B., Tseregounis, S., and Ying, L. H., "Cold Starting and Pumpability Studies in Modern Engines - results from the ASTM D02.07C Low Temperature Engine Performance Task Force Activities: Engine Selection and Testing Protocol," Oil Flow Studies at Low Temperatures in Modern Engines, ASTM STP 1388, H. Shaub, Ed., American Society for Testing and Materials, West Conshohocken, PA, 2000.

Kenneth O. Henderson,[4] Christopher J. May,[1] Enrique F. De Paz,[2] Fred W. Girshick,[3] Robert B. Rhodes,[5] Spyros Tseregounis,[6] and Lisa H. Ying[7]

Cold Starting and Pumpability Studies in Modern Engines - Results from the ASTM D02.07C Low Temperature Engine Performance Task Force Activities: Phase I Pumpability Testing

Reference: Henderson, K. O., May, C. J., De Paz, E. F., Girshick, F. W., Rhodes, R. B., Tseregounis, S., and Ying, L. H., **"Cold Starting and Pumpability Studies in Modern Engines - Results from the ASTM D02.07C Low Temperature Engine Performance Task Force Activities: Phase 1 Pumpability Testing,"** *Oil Flow Studies at Low Temperatures in Modern Engines, ASTM STP 1388,* H. Shaub, Ed., American Society for Testing and Materials, West Conshohocken, PA, 2000.

Abstract: LTEP Phase 1 pumpability testing focused on overnight cooling evaluation of LTEP 1-7 reference oils. Generally, each engine/oil combination was cooled in 16 hours to the desired test temperature before motoring for the pumpability evaluation. All tests in which limiting pumping criteria was achieved indicated failure by flow limited behavior rather than by air-binding failures.

Two approaches were investigated for relating the time to attain a pressure at two specified engine locations to the lubricant's properties. One was to develop correlations directly between pressurization time and the lubricant's temperature in the sump, and then use these correlations to calculate the minimum pumping temperature and the corresponding maximum pumpable viscosity. The other was to develop correlations between pressurization time and the lubricant's viscosity.

The first approach compared times to reach a given pressure after the oil pump (Pump Out) or the oil distribution passage downstream from the filter (Near Galley) for the observed lubricant sump temperature. A first-order exponential decay was found to give the best overall correlation for all the engine/oil combinations. All four test engines

[1] Senior Research Associate, Imperial Oil, Research Dept., P.O. Box 3022, Sarnia, Ont., Canada N7T 8C8.
[2] Project Engineer, Consumers Union, 101 Truman Avenue, Yonkers, NY 10703-1057.
[3] HD Formulation Science Leader, Infineum USA LP, P.O. Box 536, Linden, NJ 07036.
[4] Vice President of Technology, Cannon Instrument Co., P.O. Box 16, State College, PA 16804-0016.
[5] Consultant, Infineum, USA LP, 11735 Normont Dr., Houston, TX 77070.
[6] Staff Research Engineer, General Motors Research Laboratories, P.O. Box 9055, Warren, MI 48090-9055.
[7] Senior Statistician, Infineum USA LP, P.O. Box 536, Linden, NJ 07036.

exhibited a dependency of Near Galley pressurization time on sump temperature, except for combinations involving LTEP 1 oils and the 4.6 L engine, where data were limited by cold room capabilities. Near Galley pressurization was also found to be a more stringent criterion than Pump Out in most cases. Using a 60 second 10 kPa limit, minimum pumping temperatures (MPTs) were calculated for each engine/oil combination, along with a certainty level based upon degree of extrapolation. Based upon these MPTs, limiting ASTM D 3829 viscosity was approximately 93 Pa·s. The results also indicated that, as in the startability studies, the viscosity limit for the 4.0 L I6 engine was compatible with those of the December 1994 SAE J300 Viscosity Classification Specification, while the other engines were more in line with the December 1995 J300 Viscosity Classification Specification limit.

In the second approach to pumpability analysis, a correlation between oil viscosity and pressurization time for each engine at the Pump Out or Near Galley location was developed. A linear model relating lubricant viscosity to pressurization time was found to be adequate. Analysis was done to compare viscosities as measured by ASTM Test Method for Predicting the Borderline Pumping Temperature of Engine Oil (D 3829), Test Method for Determination of Yield Stress and Apparent Viscosity of Engine Oils at Low Temperature (D 4684) and Test Method for Low Temperature, Low Shear Rate, Viscosity/Temperature Dependence of Lubricating Oils Using, a Temperature-Scanning Technique (D 5133). The model equations provided tools to calculate the limiting viscosity for these engines. As with the first approach, the results indicated that the viscosity limit for the modern engine designs was more in line with the current April 97 J300 Viscosity Classification Specification limit.

Additional work was conducted to model the time/oil pressure curves from the LTEP work. It was found that most of the pressurization curves could be modeled well using a logit function.

Keywords: light-duty engines, mini-rotary viscosity, scanning brookfield, low temperature pumpability

Introduction

This paper provides a discussion of the Phase I low temperature pumpability studies conducted by the Low Temperature Engine Performance (LTEP) task force. Details on this activity and discussions of other aspects are available in the references [1– 6].

The Phase I portion of the pumpability study focused on overnight cooling of the engine. Generally, each engine/oil combination was cooled in 16 hours to the desired test temperature before motoring for the pumpability evaluation.

For these studies all engines were equipped with temperature sensors in at least three sump locations. These were near the top of the oil, in the middle, and near the bottom close to the oil pickup. These locations are described in more detail in the test protocol section of the research report [1].

The LTEP oils 1 through 7 were all commercial engine oils meeting SAE J300 Specifications at the time of selection. The viscometric characteristics of these oils are detailed in the research report and discussed in another portion of this symposium [1,3].

This review will focus on data collected in four engines. These are the 2.2 L, 3.8 L

4.0 L and 4.6 L engines. Each engine/oil combination was evaluated for pressurization time at the Minimum Starting Temperature (MST), 3 degrees below MST and 6 degrees below MST. If an engine/oil combination failed to pressurize in the allotted time at MST then the next test was run 3 degrees above MST.

The engines were motored to ensure a consistent operating regime as the pumpability studies were conducted at or below the engine's minimum starting temperature. Two general configurations were used for the engine pumpability studies which are: 1) an engine mounted on a test stand that was directly turned by a frequency controlled motor, or 2) a vehicle-mounted engine that was turned through the drive train by placing the vehicle on a chassis dyno. The second configuration required a vehicle with a manual transmission. The engines evaluated in these pumpability studies were a subset of those used in the startability portion of the program. This was partially a reflection of the fact that all engines were not available with manual transmissions. A full listing of participants and engines evaluated in the pumpability portion of the program is detailed in the research report [1].

As a result of the differences between test sites in the way the engines were motored, a time period of 10 seconds was set for reaching the published fast idle speed. This is noted in Appendix E of the research report [1].

Motoring the engines lowers the minimum temperature at which the engine/oil combinations can be evaluated. This permits testing below the minimum start temperatures (MST) as determined in the startability portion of this program. We should not forget that the MST was determined at ideal conditions for starting: Essentially unlimited battery power, and a freshly tuned ignition system as noted in the protocol. The effect of fuel dilution and resultant viscosity reduction on the pumpability observations is removed by motoring the engine. Thus, the pumpability observations described below are possibly beyond the operating limit in the sense that for them to be matched in the field, would require special efforts or extremely unusual circumstances.

Figure 1 is representative of a 16-hour cooling regime to program a nominal -35°C pumpability evaluation temperature. This figure indicates the lag between air temperature and oil sump temperature. The sump temperature shown is from the temperature probe located at the oil's mid-height level in the oil pan. Although not shown, the other oil temperatures deviate slightly from the one shown by a degree or two, but after about 12 hours these deviations drop to be in the tenths of a degree range.

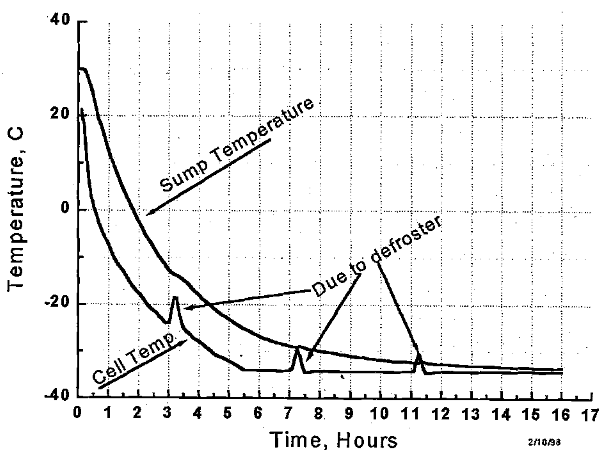


Figure 1 - *Overnight 16-Hour Cooling Profile*

A review of the data obtained from the sump temperature measurements during pumpability testing showed that the temperature rose quite rapidly at the upper and mid oil pan temperature locations. These locations were directly over the oil pickup screen. At other locations in the pan the temperature rise took much longer. The rate of temperature rise above the oil pickup screen was very similar regardless of the evaluation temperature. It was observed by one researcher that small changes in the position (1 cm) of a sensor around the oil pickup would cause a large change in observed temperature (10 °C). This is believed to be the result of the oil being in laminar flow with the cold and warm oil having a very large difference in apparent viscosity. This large difference in viscosity minimizes mixing and results in large isothermal gradients. These effects combine causing the oil to develop a channel narrow flow pattern through the sump resulting in the rapid rise in temperature of the circulated oil. If the engine continues to operate long enough the flow channel widens and the remaining oil in the oil pan warms to operating temperatures. At these test temperatures the oil directly above the oil pickup would rise 20 °C in less than 4 minutes and in 25 minutes of motored operation the oil in the sump would be at operating temperate. The rapid rise in oil temperature is possible attributable to energy put into it by the oil pump and the heat absorbed as it flows through the high load areas of the engine. All of these data emphasize how dynamic this whole process is, for once the engine begins to turn, the oil and engine temperature begin to rise immediately. We have presumed that we can model the system by using the initial sump oil temperature as constant reference point and monitoring the oil pressure rise with time.

Terminology

The following terms are used throughout the rest of the report.

Initial Pressurization Time (IPT) - The elapsed time from the time the crankshaft starts turning until the recorded pressure is first attained.

Sustained Pressurization Time (SPT) - The elapsed time from the time the crankshaft starts turning until the recorded pressure is maintained. In some tests after the IPT was reached, the pressure would fall back to an intermediate pressure, then rising to the pressure of interest.

Pump Out - The pressure location between the pump outlet and the filter inlet.

Pump Out Pumpability Limit - The pressure at the Pump Out must reach 150 kPa in a maximum of 15 seconds.

Near Galley - This is the galley supplying oil to the cam bearings that is nearest the oil filter. A V6 or V8 engine usually has two galleys supplying the cam bearings.

Near Galley Pumpability Limit - The pressure at the Near Galley must reach 10 kPa in a maximum of 60 seconds.

Nominal Value - The time used (60 seconds) to indicate in the data that a pumpability test was started but aborted because the pressure at the Near Galley still had not reached the required 10 kPa after 60 seconds.

Lowest Observed Pressurization Temperature - The lowest test temperature at which the acceptable pumpability criteria were met.

Minimum Pumping Temperature - The calculated minimum temperature at which the individual pumpability limits would be met.

Pressurization Time Correlations

Two approaches were used to relate the time to attain pressure at the two pumpability limit locations to the lubricant's properties. Although pressurization time data were collected at a number of locations in the engine, the focus of the data analysis was at the Pump Out and Near Galley locations. One analysis approach was to develop correlations between pressurization time and lubricant viscosity. The other approach was to first develop correlations between pressurization time and lubricant temperature in the sump, and then use these correlations to calculate the minimum pumping temperature and the corresponding maximum pumpable viscosity. These two approaches are summarized in the following discussion. All of the data collected are contained in the research report. [1]

Pressurization Time vs. Lubricant Temperature

The first view of the data will compare time to reach a given pressure at the Pump Out or Near Galley for the observed lubricant sump temperature. Only four of the seven engines were tested at multiple temperatures on each oil. These were the 2.2 L, 3.8 L, 4.0 L, and 4.6 L engines. This analysis focuses on pressurization time at either the Pump Out or Near Galley location for these four engines.

After evaluating several ways of fitting the data, a first order exponential decay was found to give the best (highest R^2) overall correlations for all the engine/oil combinations. The general form of the equation is:

$$SPt = SPt_0 + a\ e^{((t0-t)/b)}$$

where in this case t is the lubricant temperature in the sump in Kelvin and SPt is the time for sustained pressure in seconds. SPt_0, t0, a, and b are constants determined from curve fitting of the data and are dependent on the oil, the engine, the pressure location and the pumpability criteria. The analysis program set the initial value of t0 to a value close to the minimum value of t in the data set. The initialization value of the SPt_0 is set to a number close to the asymptotic value of SPt for large t values.

Evaluation of Pump Out Data by Engine

Data for the 2.2, 3.8, 4.0 and 4.6 L engines, were plotted for evaluation of the relationships between the time to reach 150 kPa at the Pump Out location and the engine sump temperature for each of the LTEP 1 thru 7 oils.

The data and regression lines for the 3.8 L engine are shown in Figure 2. Although not shown a similar view is seen with the 2.2 L data. The data set for the 3.8 L engine contains four data points in excess of 60 seconds indicating the lubricant was too viscous to flow. These are noted as nominal values in the figure. In the Pump Out data set for the 2.2 L engine, one LTEP 3 and one LTEP 5 data points are grossly inconsistent with the other 3 data points for that oil. These apparent outliers have been excluded from the Pump Out regression analysis. In these figures the average minimum start times are included for comparison and indicated by the straight vertical dotted line connected to the labeled box.

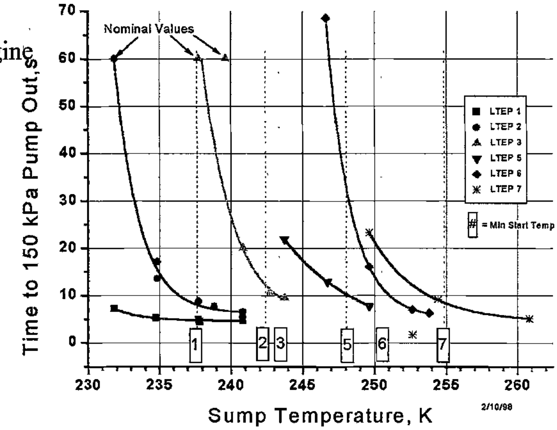


Figure 2 - *Pump Out 3.8 L V6 Engine*

The coefficients of determination for the 2.2 and 3.8 L engine/oil combinations at the Pump Out location were all greater than 0.92. Only LTEP 5 and 6 with the 2.2 L engine have a smaller coefficient of determination but they are however still greater than 0.75.

The data sets for the 4.0 and 4.6 L engines showed little increase in pressurization time as the sump temperature declines. All of the pressurization times were well below the limiting criteria. As a result it was not possible to determine correlations that could estimate the time to reach the limiting criteria. This lack of correlation between pressurization time and temperature was possibly due to the infrequency of data collection (two readings per second).

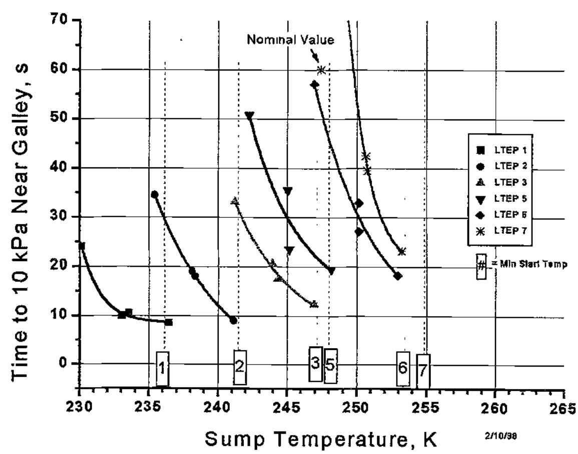


Figure 3 - *Near Galley 2.2 L I4 Engine*

Evaluation of Near Galley Data by Engine

In a similar fashion, reviewing the data on the 2.2, 3.8, 4.0 and 4.6 L engines at the Near Galley location, we find nearly all the engine/oil combinations show a relationship between pressurization time and sump temperature. Figures 3, 4, and 5 show the relationship between the time to reach 10 kPa at Near Galley location and the engine sump temperature for each engine/oil combination and its corresponding regression line. All four engines exhibited a dependency of pressurization time on sump temperature, except LTEP 1 in the 4.6 L engine. A relationship could not be determined as this engine/oil combination was essentially run at only one temperature because of cold room capability.

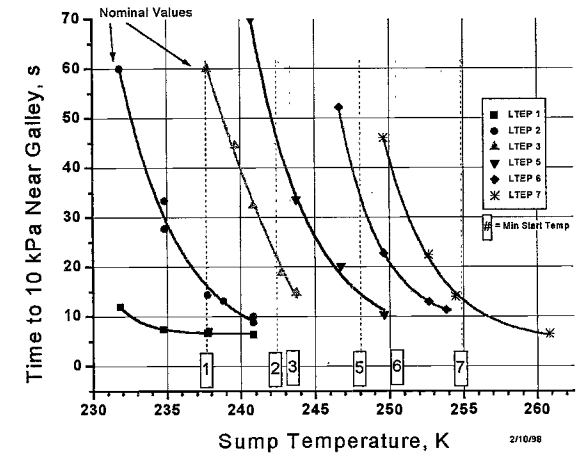


Figure 4 - *Near Galley 3.8 L V6 Engine*

At the Near Galley location all of the values for coefficient of determination (R^2), except two, were greater than 0.93. The 2.2 L engine with LTEP 5 still had a fairly high 0.89 R^2, while the 4.6 L engine had a significantly lower 0.60 R^2, again with oil LTEP 5.

LTEP 5 with the 4.6 L engine appeared to behave most peculiarly. While at the Pump Out location (1) it showed poorer performance than LTEP 3 (as one would expect), at the Near Galley, Figure 5 it performed better. We know of no viable explanation why this reversal takes place and it is the only case where W grade reversal was observed.

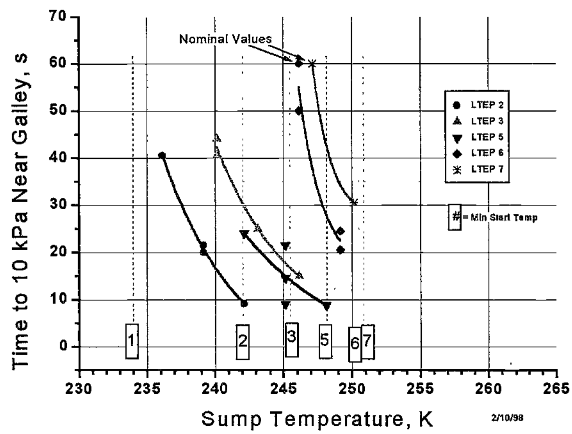


Figure 5 - *Near Galley 4.6 L V8 Engine*

Phase 2 Oils under Phase 1 Conditions

Pump Out — LTEP 22, 23, 24 and 26 oils were subjected to Phase 1 16-hour cooling conditions in two of the engines in the program. This SAE 5W-30 (23 & 24) and SAE 10W-30 (22 & 26) oils exhibited yield stress to some degree in pumpability bench tests. Comparing the Series 20 data to LTEP 2 (5W) and 3 (10W) shows they perform in the engine in a nearly identical manner over the range of temperatures tested[1]. At the Pump Out location with the 2.2 L engine these oils did show a dependency of pressurization time on sump temperature in these tests as was seen earlier with LTEP 2 and 3. The dependency of pressurization time on sump temperature in the 4.6 L engine was again not present. This is consistent with earlier observations on LTEP 2 and 3 with this engine (Figure 6).

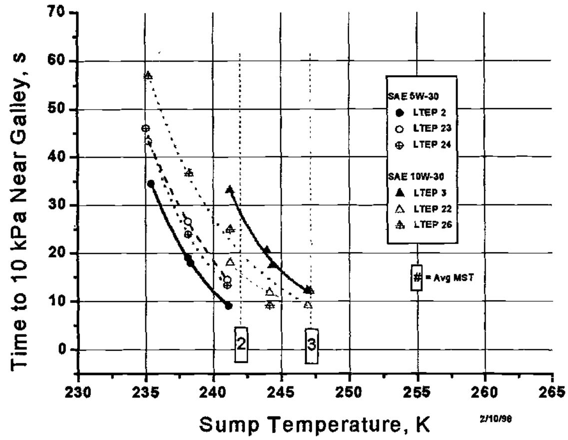


Figure 6 - *Near Galley 2.2 L I4 Engine, 20 Series*

The Near Galley location data shown in Figures 6 and 7 for the 2.2 L and 4.6 L engines respectively indicate a dependency of pressurization time on sump temperature similar to LTEP 2 and 3. Again, generally speaking, all the correlations have an R^2 >0.99, with the exception of oils 22 and 26 at the Near Galley with the 2.2 L engine.

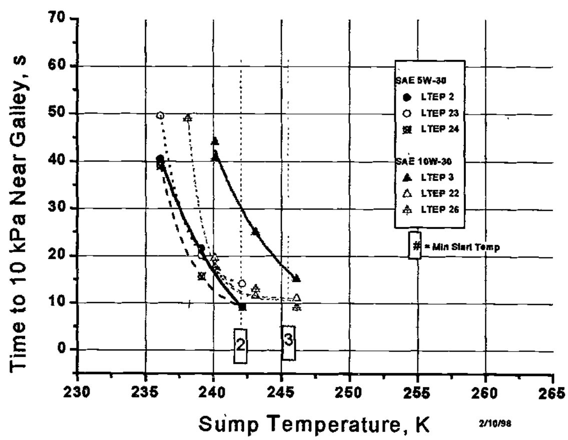


Figure 7 - *Near Galley 4.6 L V8 Engine, 20 Series*

Pressurization Time - Limiting Criteria

A survey of the engine manufacturing industry at the beginning of this study indicated that in order to protect an engine, an oil needed to reach a set pressure at the filter (Pump Out) or the galley (Near Galley) in a specified time. For the Pump Out location the specification was for a sustained pressure of 150 kPa in a maximum of 15 seconds. At the Near Galley the specification was for a sustained pressure of 10 kPa in a maximum of 60 seconds. For this study then the Lowest

Observed Pressurization Temperature (LOPT) is at the lowest sump temperature tested when the pressurization time satisfies the above specification criteria. A listing of the LOPTs for each engine and oil at these specific conditions for Pump Out and Near Galley respectively are contained in Tables 1 and 2. The "<" marks indicate engine/oil combinations where the lowest temperature tested was not low enough to achieve or exceed the pressure/time criteria. In these cases, the engine/oil combination would need to be tested at lower temperatures in order to determine exactly where they would exceed these criteria.

Table 1 - *Pump Out*
Lowest Observed Pressurization Temperature, Celsius,
for 150 kPa Pressure in ***less than*** *15 seconds*

LTEP Oil	1	2	3	5	6	7
2.2 L I4 Engine, °C	**<-43.0**	**<-37.7**	**<-31.9**	**<-30.9**	**<-26.2**	**<-25.7**
3.8 L V6 Engine, °C	**<-41.3**	-38.2	-31.5	**<-29.4**	-23.4	**<-23.5**
4.0 L I6 Engine, °C	**<-39.7**	**<-37.0**	**<-31.3**	**<-28.6**	**<-22.6**	**<-20.2**
4.6 L V8 Engine, °C	**<-42.0**	**<-37.0**	**<-33.0**	**<-31.0**	**<-27.0**	-24.5
4 Engine Average, °C	**<-41.5**	**<-37.5**	**<-31.9**	**<-30.0**	**<-24.8**	**<-23.5**

* For clarity, less than temperatures are printed in bold

In Table 1 the sump temperatures listed for the 2.2, 4.0 and 4.6 L engines are the lowest temperatures at which the oil was tested. The experimental design called for evaluating the oils at 0, 3 and 6 °C below the minimum start temperature for that engine. For these three engines, the oils were not evaluated at a temperature low enough to reach the limiting criteria. So the temperature to reach 150 kPa at the Pump Out in 15 seconds is at a temperature lower than that shown in the table. However, for the 3.8 L engine the data did traverse the 15 second time for some of the oils and the LOPT values were interpolated from the appropriate regression model.

Table 2 - *Near Galley*
Lowest Observed Pressurization Temperature, Celsius
for 10 kPa Pressure in ***less than*** *60 seconds*

LTEP Oil	1	2	3	5	6	7
2.2 L I4 Engine, °C	**<-43.0**	**<-37.7**	**<-31.9**	**<-30.9**	**<-26.2**	-23.5
3.8 L V6 Engine, °C	**<-41.3**	-41.4	-35.5	-31.8	**<-26.5**	**<-23.5**
4.0 L I6 Engine, °C	**<-39.7**	-36.7	-31.0	-28.7	-22.6	-20.2
4.6 L V8 Engine, °C	**<-42.0**	**<-37.0**	**<-33.0**	**<-31.0**	-27.2	-21.4
4 Engine Average, °C	**<-41.5**	**<-38.2**	**<-32.8**	**<-30.6**	**<-25.6**	**<-22.2**

* For clarity all less than temperatures are printed in bold

In Table 2 the sump temperatures listed for the Near Galley location is either the lowest temperature at which the oil was tested or the temperature at which the limiting pressure/time criteria were achieved. Compared to the Pump Out location, where only 4 of 24 engine/oil combinations data spanned the limiting criteria, 11 of 24 data combinations spanned the limiting criteria for the Near Galley. Since the test temperatures were the same for both cases, this indicates that the Near Galley criterion is the more stringent for most engines.

Table 3 - *Pump Out*
Minimum Difference between Minimum Start Temperature and Lowest Observed Pressurization Temperature, Celsius

LTEP Oil	1	2	3	5	6	7	Average
2.2 L I4 Engine, °C	>6.0	>6.0	>5.9	>5.9	>6.5	>7.4	>6.3
3.8 L V6 Engine*, °C	>4.7	7.4	1.7	>4.3	0.7	>5.2	>4.0
4.0 L I6 Engine, °C	>9.5	>8.5	>8.2	>10.6	>10.7	>13.5	>10.2
4.6 L V8 Engine, °C	>2.8	>5.9	>5.3	>5.8	>4.4	>2.2	>4.4
4 Engine Average, °C	>5.8	>7.0	>5.3	>6.7	>5.6	>7.1	

* 3.8 L Engine MST is an average of 2 engines; LOPT data is from a single engine.

Tables 3 and 4 for Pump Out and Near Galley locations respectively show the difference between minimum start temperature and the LOPT for each engine/oil combination along with the overall average difference for each oil and for each engine.

Table 4 - *Near Galley*
Minimum Difference between Minimum Start Temperature and Lowest Observed Pressurization Temperature, Celsius

LTEP	1	2	3	5	6	7	Average
2.2 L I4 Engine, °C	>6.0	>6.0	>5.9	>5.9	>6.5	5.2	>5.9
3.8 L V6 Engine*, °C	>4.7	10.6	5.7	6.7	>3.8	>5.2	>6.1
4.0 L I6 Engine, °C	>9.5	8.2	7.9	10.7	10.7	13.5	>10.1
4.6 L V8 Engine, °C	>2.8	>5.9	>5.3	>5.8	4.6	0.9	>3.9
4 Engine Average, °C	>5.8	>7.7	>6.2	>7.3	>6.4	>5.8	

* 3.8 L Engine MST is an average of 2 engines; LOPT data is for a single engine.

For the Pump Out location the LOPT is 0.7 to 13.5 °C below the MST, with the overall average for each of the oils being greater than 5 °C below the corresponding MST. The averages for each engine are all over 4 °C below the MSTs. Looking at the minimum differences at the Pump Out location (Table 3) by engine shows the 3.8 L has the smallest average difference between MST and LOPT while the 4.0 L has the largest difference. The small difference for the 4.6 L engine can be attributed to the results with LTEP 1 and 7. However, the failure temperature for 58% of the engine/oil combinations is still more than 6 °C below the Minimum Start Temperature for that particular oils W-grade.

The Near Galley location indicates similar difference between these MSTs and LOPTs. At the Near Galley location (Table 4) the smallest difference shifts to 4.6 L engine. In this case the shift to this engine can be attributed to LTEP 7's performance and to some extent LTEP 1's performance.

Limiting Viscosity

The previous section established temperatures below the minimum start temperature where these engines have demonstrated the ability to meet the two pumpability criteria with each of the oils. In most of the cases (20 of 24 at the Pump Out and 19 of 24 at the Near Galley) however, the results indicated that the limiting pumpability criteria would be met only at a temperature lower than the lowest tested temperature. To determine the maximum acceptable oil viscosity for each of these engines with each of these oils, further testing would be needed at lower temperatures. Since this was no longer an option, the question arose as to how the available information could be used to estimate the maximum oil viscosity that would satisfy the pumpability criteria for these engines.

One approach is to use only the data from engine/oil combinations where the data collected span the limiting criteria, thus allowing the data value to be interpolated. However, this approach would quickly eliminate 75% of the engine/oil combinations used in this program. This would bias the conclusions to those engine/oil combinations and not make them generally applicable to the rest of the engines and oils in this program or to those in the marketplace. To circumvent this shortcoming, an alternative approach was developed.

This alternate approach involved calculation of the predicted minimum pumping temperature (MPT) at the limiting time to pressure criteria (15 seconds at Pump Out and 60 seconds at Near Galley) using the exponential decay correlations previously developed. Since 75% of these results will be extrapolations, the extent of extrapolation was used to assign a 'certainty' level to each calculated result. The certainty level was calculated (as percent) by determining how close the lowest test temperature for a particular engine/oil combination was to the time limit. For example, at the lowest tested temperature (-39.7 °C), the LTEP 1 with 4.0 L combination reached 150 kPa limit at the Pump Out in 1.5 seconds. This translates to a 10% ((1.5 s/ 15 s) x 100) certainty level. On the other end, the LTEP 6 with 2.2 L combination at the Near Galley required 57 seconds to reach the 10 kPa limit, or a 95% ((57 s/ 60 s) x 100) certainty level. The certainty level for any engine/oil combination which reached the limit during testing was automatically assigned a 100% certainty level. The Phase 2 oils were excluded from this analysis because they were tested in only two of the engines.

Table 5 - *Pump Out*
Calculated Minimum Pumping Temperature, °C

LTEP Oil	1	2	3	5	6	7
2.2 L I4 Engine	-43.1	-59.3	-64.1	-42.2	-32.3	-29.5
3.8 L V6 Engine	-44.1	-38.2	-31.4	-27.2	-23.3	-21.6

Table 6 - *Pump Out*
Certainty Level = Percent of Limit

LTEP Oil	1	2	3	5	6	7
2.2 L I4 Engine	49	31	22	22	35	57
3.8 L V6 Engine	50	100	100	100	100	100
4.0 L I6 Engine	10	25	10	39	28	11
4.6 L V8 Engine	-	39	47	50	35	40
4 Engine Average	39	49	45	53	50	52

Table 5 shows the predicted MPT and Table 6 shows the certainty levels for the Pump Out criteria. MPTs for the 4.0 L and 4.6 L engines could not be calculated because a correlation could not be developed. Note that the calculated MPT's for the two engines are completely different from each other except for LTEP 1. Note also that the certainty levels for the 2.2 L engine are also very low (22 to 57%), while those for the 3.8 L are 100% except for LTEP 1. The certainty levels for the two other engines used in the program are also very low (10 to 50%), which makes the overall average certainty level for the Pump Out location also quite low (39 to 53%). The low certainty and the inability to generate useful correlations for two of the four engines, makes it in appropriate to estimate an overall viscosity limit for the engine/oil combinations used in this program based on Pump Out criteria.

Table 7 - *Near Galley*
Calculated Minimum Pumping Temperature, °C

LTEP Oil	1	2	3	5	6	7
2.2 L I4 Engine	-44.7	-39.7	-34.7	-31.7	-26.4	-23.4
3.8 L V6 Engine	**-45.2**	**-41.3**	**-35.4**	-31.8	-26.9	-24.5
4.0 L I6 Engine	***-41.1***	***-36.5***	***-31.1***	***-28.6***	***-22.6***	***-20.2***
4.6 L V8 Engine		-38.9	-35.0	**-37.5**	**-27.2**	**-26.0**
Average MPT with	-43.7	-39.1	-34.1	-32.4	-25.8	-23.5
Std Dev with 4.0 L	2.2	2.0	2.0	3.7	2.1	2.5
Average MPT without	-45.0	-40.0	-35.0	-33.7	-26.8	-24.6
Std. Dev. without 4.0	0.4	1.2	0.4	3.3	0.4	1.3

Bold = Lowest MPT: ***Bold Italics = Highest MPT***

At the Near Galley location, the results of this analysis are much more promising. Tables 7 shows the predicted MPT while Table 8 shows the certainty levels for the Near Galley criteria. The numbers in bold indicate the engine with the lowest (coldest) MPT for that particular oil, and the numbers in italics indicate the combination with the highest MPT's. The 4.0 L engine has the highest MPTs for all the oils. This is the same trend shown by this engine in the startability studies. [1,4] Glancing down any column on this table, it is quickly visible that the 4.0 L result is typically 3 to 4 °C higher than the rest of the engines.

As with the startability studies, the 4.0 L engine appears to give significantly different results from the rest of the engines. This is clearly evident from the effect that its results have on the average MPT for each oil and its standard deviation. By including the 4.0 L engine, the average MPT is raised by 1 to 2 °C. More importantly the standard deviation is reduced, on average, by 2.4 to 0.6 °C by excluding the 4.0 L engine. The fact that the 4.0 L engine is totally different from the other engines in both its starting and pumping characteristics is clearly visible in Figure 8 which plots MSTs and the MPT's for the 4.0 L and the other engines. Applying the statistical t-test to these MPT values for the four engines show the 4.0 L engine to be significantly different from the other three engines. The average MST's is shown for the other engines. It was determined in Section 3.2 that the MST's of the other engines are not statistically different.

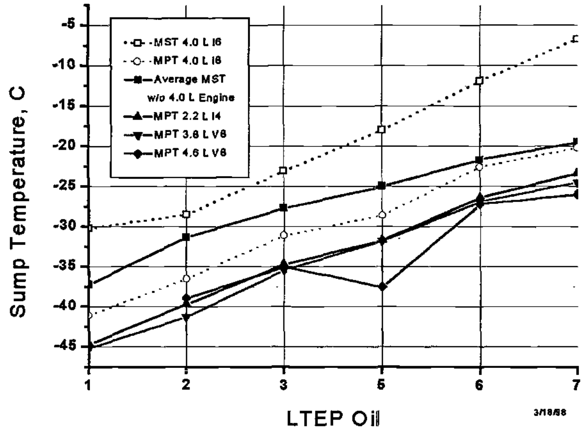


Figure 8 - *Average MST vs MPT*

Table 8 - *Near Galley*
Certainty Level = Percent of Limit (60 seconds)

LTEP Oil	1	2	3	5	6	7
2.2 L I4 Engine	40	57	55	85	95	100
3.8 L V6 Engine	20	100	100	100	87	77
4.0 L I6 Engine	48	100	100	100	100	100
4.6 L V8 Engine		68	73	40	100	100
Average	36	81	82	81	95	94

The Near Galley engine criterion, when applied to the 3.8 L and 4.0 L engines, appears to predict when an absence of pressurization will occur. Whenever tests were run at or below the calculated minimum pumping temperatures listed in Table 7, the pressurization time traces generated in the near galley of both the 3.8 L and 4.0 L engines showed an absence of pressure generation through at least a 60-second time period. During this time period the oil pump inlet indicated a negative pressure (vacuum), thus implying the oil inlet tube was filled with oil.

Since the MPT's of all the engines, except the 4.0 L, is not significantly different from one another, the average MPT for each oil may be used to represent all the engines. Thus, the overall results for the MST and MPT are shown in Figure 9. These show that the safety margin between the minimum starting temperature and the minimum pumping temperature is significantly higher for the 4.0 L engine (ranging from 8 to 13.5 °C) than in the rest of the engines (5.1 to 8.7 °C). This difference appears to be due mainly to the much higher minimum start temperatures of the 4.0 L engine.

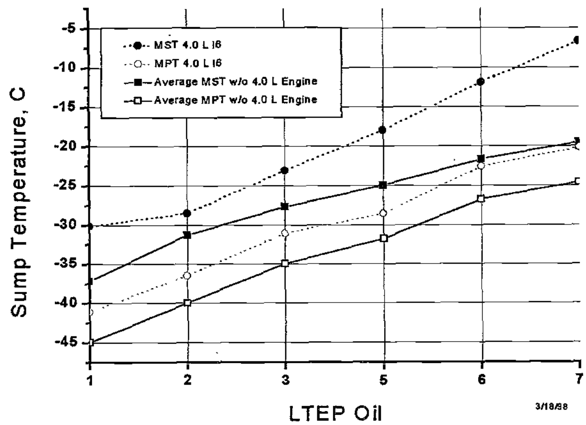


Figure 9 - *Average Engine vs 4.0 L I6 Engine*

Looking at the certainty level of the results at the Near Galley (Table 8) shows that all the engines give a low certainty level with LTEP 1 (20 to 48%). This can be attributed to the lack of cooling capacity in the cold rooms used for these studies to achieve operational temperatures appropriate for SAE 0W oils. The certainty level then generally increases (57 to 100%) for all engines as the oil grade increases (except for the 4.6 L engine with LTEP 5). Averaging the certainty level for each oil shows that, on average, the certainty level is high (81 to 82%) for LTEP 2 to LTEP 5 and very high (94 to 95%) for LTEP 6 and 7. This suggests that the estimated MPT for oils LTEP 2 to LTEP 7 are probably very reliable estimates, while those for LTEP 1 should be considered suspect.

Table 9 - *Near Galley*
Calculated Viscosity at Average MPT
Excludes 4.0 L I6 Engine

LTEP Oil	1	2	3	5	6	7	LTEP 2 - 7 Average	Std. Dev.
D 3829 Viscosity, Pa·s	41.6	76.8	71.3	131.3	74.2	97.0	90.1	25.2
D 4684 Viscosity, Pa·s	32.2	55.3	104.0	167.3	133.5	121.3	116.3	41.2
D 5133 Viscosity, Pa·s	35.1	64.1	70.7	497.6	/	68.0		

Note: Extrapolated values are underlined.

The estimated MPT's were used in the appropriate temperature viscosity correlations to calculate each oil's maximum pumpable viscosity. The average MPTs were used for the three engines (2.2 L I4, 3.8 L V6, 4.6 L V8), and the individual MPTs were used for the 4.0 L engine. The viscosities as measured by the methods D 3829 (Original MRV), D 4684 (MRV TP1) and D 5133 (Scanning Brookfield) are shown in Tables 9 and 10 along with each method's average across all the oils and its corresponding standard deviation. The underlined values were obtained from the correlation equation by extrapolation. The extrapolation value lies either outside of the viscosity data collected with these test methods or outside the range of the test method. D 5133 cannot measure viscosities in excess of about 45 Pa·s. These results show that most of the D 5133 viscosities are beyond the capability of the instrument, and those for LTEP 1 are outside the capabilities of all the test procedures.

Table 10 - *Near Galley*
Calculated Viscosity at Average MPT
for 4.0 L I6 Engine

LTEP Oil	1	2	3	5	6	7	LTEP 2 - 7 Average	Std. Dev.
D 3829 Viscosity, Pa·s	19.5	31.0	35.5	46.7	39.0	40.4	38.5	5.8
D 4684 Viscosity, Pa·s	17.6	24.8	37.4	109.0	81.2	53.6	61.2	34.0
D 5133 Viscosity, Pa·s	20.1	47.5	33.0	103.6		52.3		

Note: Extrapolated values are underlined.

Concentrating on the valid results, one sees that the D 4684 (MRV TP1) results are generally higher and more inconsistent (higher standard deviation across the oils) than those for D 3829. This should be expected because the cooling cycle for D 4684 method was designed to hold the oil in the cloud point region for an extended period of time to enhance the formation of a flow impeding wax structure. Since the cooling cycle of the D 3829 more closely resembles that used in the engine testing, one would expect to find the best correlation with these viscosity results. This is in fact true for both the 4.0 L and the other engines. Again omitting the results for LTEP 1, the D 3829 viscosity for the average engine ranges from 71.3 to 131.3 Pa·s, with an average of 90.1 and a standard deviation of 25.2 Pa·s. These results suggest that, as in the startability studies, the viscosity limit for 4.0 L engine is compatible with the viscosity limits (30.0 Pa·s) from the December 1994 SAE J300 Viscosity Classification Specification (Table 10), while the other engines are more in line with the December 1995 J300 Viscosity Classification Specification limit of 60.0 Pa·s (Table 9).

For a number of tests, an MRV was placed in a test cell during the engine pumpability test. The test oil was placed in at least three of the MRV test cells. At the conclusion of an engine pumping test, the MRV test temperature was recorded and the viscosity determined. In Table 11 the viscosities determined in the test cell MRV are compared to the interpolated D3829 data. Linear regression of the test cell MRV viscosity data verus the D 3829 calculated data yields an R^2 value of 0.9295. These observations provide additional support that the ASTM D 3829 method is appropriate for estimation of Phase 1 pumping time versus sump viscosity relationships.

Pressurization Time vs Lubricant Viscosity

The time to reach a pressure at any location in an engine is affected by many differences in the lubricant flow system designs. These include filter porosity, filter bypass valves, number of bends, a variation in size of passages, etc. However, for a given engine, the time to reach a pressure should show a strong relationship to the lubricant's viscosity.

Table 11
Test Cell MRV Viscosity versus Calculated D 3829 Viscosity

LTEP Oil	Test Cell MRV Temperature, °C	Test Cell MRV Viscosity, Pa·s	Interpolated Viscosity, Pa·s
2	-35	29.1	22.1
2	-38	42.4	44.8
2	-40	83.1	76.8
3	-30.7	33.2	33.0
3	-33.6	68.7	55.7
5	-23.4	18.8	18.7
5	-26.3	31.4	30.7
5	-28.4	56.8	45.0
5	-32.2	102.5	99.3
6	-19.4	22.5	24.3
6	-20.6	28.1	29.0
6	-23.4	46.5	43.9
6	-26.6	84.2	71.9
7	-12.8	10.4	11.8
7	-18.6	25.9	30.2
7	-20.8	37.3	45.2
7	-23.8	68.5	81.9

In this portion of the analysis a correlation between viscosity and pressurization time for each engine at the Pump Out or Near Galley location was developed. The viscosity values were based on the sump temperature at the start of the pumpability test. In an earlier section of this report, the determination of the viscosity/temperature equations was discussed. These equations were used to calculate the lubricant's viscosity at the start of the test. During this evaluation, a comparison was made between initial pressurization time and viscosity as well as sustained pressurization time. Analysis using the initial pressurization time was not progressed, as the data analysis group felt the time to reach a sustained pressure was more significant for engine operation.

A linear model relating lubricant viscosity to pressurization time was found to be adequate. The equation takes the following form:

$$SPt = SPt_i + d\ (Viscosity)$$

where SPt is the time to sustained pressure, SPt_i is the intercept with the x axis and d is the rate of change in terms of time and viscosity.

Pump Out Pressurization

The effect of D 3829 viscosity on sustained pressurization time is shown by the data for the 2.2 L engine with LTEP oils 1 through 7 and 22 through 26 in Figure 10. This figure includes the correlation line from the regression calculation. Data for the 3.8 L engine with LTEP 1 through 7 oils yields a similar relationship. These regression calculations did not include any nominal values. A similar analysis was done for SPt versus viscosity, where the viscosity was obtained by D 4684 and D 5133. Coefficient of determination for D 3829 viscosity is larger in both engines than that determined by the other test methods. This is consistent with the idea that the cooling program for the engines closely resembles that used in D 3829. It is also consistent with the idea that D 4684 was designed to transit the cloud point region slowly to maximize wax structure formation. The D 5133 data set contains fewer data points as the test method has a maximum viscosity it can measure due to the torque range of the viscometer, which is on the order of 45 Pa·s.

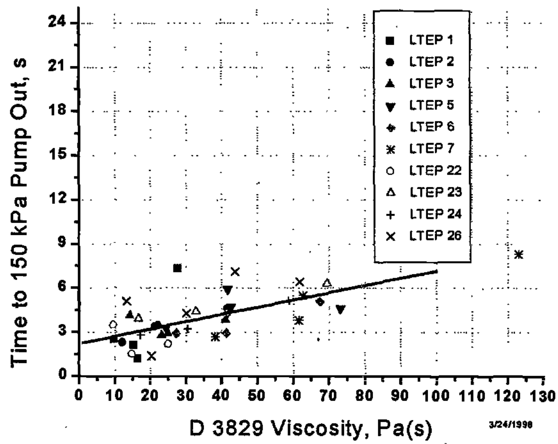


Figure 10 - *2.2 L I4 Engine*

The 4.0 L and 4.6 L engines are not included in this Pump Out analysis for the same reasons previously discussed, which is the lack of apparent dependency of pressurization time on lubricant viscosity.

Near Galley Pressurization

A comparison of D 3829 viscosity versus time to sustained pressure at the Near Galley location is shown in Figure 11 for the LTEP oils 1 through 7 in the 3.8 L engine. In the data analysis both the 2.2 L and 4.6 L engines' data analysis included the data obtained for LTEP 22 through 26. These LTEP 20 series data were collected under the same conditions as the LTEP Phase 1 oils.

Correlation equations relating viscosity to sustained pressure at the Near Galley location were calculated for each of the engines by viscosity test method. The R-square values were greater than 0.92 for the 2.2 L and 3.8 L with pumping viscosity data determined by D 3829. An example of the regression line for one engine is shown in Figure 11. As at the Pump Out location, the correlations did not include nominal values. A similar analysis was done for SPt versus viscosity where the viscosity values were obtained by D 4684 and D 5133. Again we see the coefficient of determination values are larger for the D 3829 viscosities than for the other two viscosity methods indicating a better fit. The order of R^2 values is D 3829 greater than D 4684 which is greater than D 5133. The reasons for this are the same as noted above for the Pump Out location.

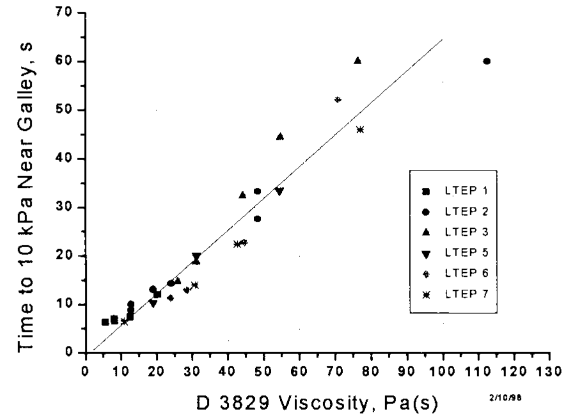


Figure 11- *3.8 L V6 Engine*

Limiting Viscosity

These model equations provide us with tools by which we can calculate the limiting viscosity for these engines. Table 12 shows the results of these calculations at the Near Galley location. The Pump Out location was omitted due to its low R^2's and the fact that the results would be based on only 2 engines.

Table 12 - *Limiting Viscosity, Pa·s*
Near Galley Location for 60 seconds to 10 kPa

Method	Engine				All Engines		4.0 L not Included	
	2.2 L I4	3.8 L V6	4.0 L I6	4.6 L V8	Average	Std Dev	Average	Std Dev
D 3829	81.8	93.0	48.3	112.2	83.8	23.2	95.6	12.6
D 4864	153.9	180.9	253.8	372.6	240.3	84.7	235.8	97.4
D 5133	213.0	164.4	74.2	1428.7	470.1	555.7	602.0	584.9

As before we see the 4.0 L I6 engine has a significant impact on the groups average, which again fits with its pedigree of being an older style engine design. These results suggest as before that, as in the startability studies, the viscosity limit for 4.0 L engines is consistent with the viscosity limits (30.0 Pa·s) from the December 1994 SAE J300 Viscosity Classification Specification, while the other engines are more in line with the December 1995 J300 Viscosity Classification Specification limit of 60.0 Pa·s.

The average D 3829 limiting viscosity, 95.6 Pa·s, for the three engines are quite similar to the limiting viscosity obtained previously (Table 9), 90.1 Pa·s, where the analysis was based on the sustained pressurization time versus sump temperature. The difference between these two approaches yields a significant difference between D 4684 average viscosity shown in Tables 9 and 12. The latter approach also increases the difference between D 3829 average viscosity and D 4684 average viscosity. The difference between D 3829 and D 4684 is reasonable in light of the fact the engines were cooled in a similar fashion to the D 3829 cooling profile and D 4684 is designed to maximize wax structure formation. This latter fact causes D 4684 viscosities to typically be larger than D 3829 viscosities. All of the viscosities indicated for D 5133 are beyond the current capability of the test method, and thus no conclusions can be drawn about the values.

The data analysis above focuses on four of the seven engines tested. When the data from all engines are combined into a single data set, the relationship between time to reach a specific pressure and pumping viscosity by D 3829 is maintained.

Conclusions

In conclusion, the time to reach a specific pressure at the Near Galley location correlates well with either sump temperature or ASTM D 3829 pumping viscosity.

It was anticipated that a better correlation would be seen at the Pump Out location between time to reach a specific pressure and lubricant properties. Since a good correlation was seen in two of the four engines, it is reasonable to attribute the lack of correlation for the other two engines to insufficient resolution in the data acquisition. Additionally the test temperature should have been extended to the point where limiting criteria were achieved at both locations. It is thought that the additional volume contributed by the distance the lubricant needs to travel plus restrictions to caused by the oil filter to reach the pressure sensing location contributed to being able to reach limiting criteria in this test protocol. It should be clear that there are significantly fewer restrictions between the Pump Out location and oil pickup as compared to the Near Galley location and the oil pickup.

A full set of conclusions for the program is in presented in reference 6.

References

[1] *Cold Starting and Pumpability Studies in Modern Engines, ASTM Research Report RR-D02-1442*, November, 1998.

[2] De Paz, E. F., Girshick, F. W., Henderson, K. O., May, C. J., Rhodes, R. B., Tseregounis, S., and Ying, L. H., **"Cold Starting and Pumpability Studies in Modern Engines - Results from the ASTM D02.07C Low Temperature Engine Performance Task Force Activities: Engine Selection and Testing Protocol,"** *Oil Flow Studies at Low Temperatures in Modern Engines, ASTM STP 1388*, H. Shaub, Ed., American Society for Testing and Materials, West Conshohocken, PA, 2000.

[3] De Paz, E. F., Girshick, F. W., Henderson, K. O., May, C. J., Rhodes, R. B., Tseregounis, S., and Ying, L. H., **"Cold Starting and Pumpability Studies in Modern Engines - Results from the ASTM D02.07C Low Temperature Engine Performance Task Force Activities: Test Oil Selection and Rheological Analysis,"** *Oil Flow Studies at Low Temperatures in Modern Engines, ASTM STP 1388*, H. Shaub, Ed., American Society for Testing and Materials, West Conshohocken, PA, 2000.

[4] De Paz, E. F., Girshick, F. W., Henderson, K. O., May, C. J., Rhodes, R. B., Tseregounis, S., and Ying, L. H., **"Cold Starting and Pumpability Studies in Modern Engines - Results from the ASTM D02.07C Low Temperature Engine Performance Task Force Activities: Cold Start Testing,"** *Oil Flow Studies at Low Temperatures in Modern Engines, ASTM STP 1388*, H. Shaub, Ed., American Society for Testing and Materials, West Conshohocken, PA, 2000.

[5] De Paz, E. F., Girshick, F. W., Henderson, K. O., May, C. J., Rhodes, R. B., Tseregounis, S., and Ying, L. H., **"Cold Starting and Pumpability Studies in Modern Engines - Results from the ASTM D02.07C Low Temperature Engine Performance Task Force Activities: Phase II Pumpability Testing,"** *Oil Flow Studies at Low Temperatures in Modern Engines, ASTM STP 1388*, H. Shaub, Ed., American Society for Testing and Materials, West Conshohocken, PA, 2000.

[6] De Paz, E. F., Girshick, F. W., Henderson, K. O., May, C. J., Rhodes, R. B., Tseregounis, S., and Ying, L. H., **"Cold Starting and Pumpability Studies in Modern Engines - Results from the ASTM D02.07C Low Temperature Engine Performance Task Force Activities: Summary and Conclusions,"** *Oil Flow Studies at Low Temperatures in Modern Engines, ASTM STP 1388*, H. Shaub, Ed., American Society for Testing and Materials, West Conshohocken, PA, 2000.

Robert B. Rhodes,[1] Enrique F. De Paz,[2] Fred W. Girshick,[3] Kenneth O. Henderson,[4] Christopher J. May,[5] Spyros Tseregounis,[6] and Lisa H. Ying[7]

Cold Starting and Pumpability Studies in Modern Engines - Results from the ASTM D02.07C Low Temperature Engine Performance Task Force Activities: Phase II Pumpability Testing

Reference: Rhodes, R. B., De Paz, E. F., Girshick, F. W., Henderson, K. O., May, C. J., Tseregounis, S., and Ying, L. H., "**Cold Starting and Pumpability Studies in Modern Engines - Results from the ASTM D02.07C Low Temperature Engine Performance Task Force Activities: Phase II Pumpability Testing,**" *Oil Flow Studies at Low Temperatures in Modern Engines, ASTM STP 1388*, H. Shaub, Ed., American Society for Testing and Materials, West Conshohocken, PA, 2000.

Abstract: The Low Temperature Engine Performance Task Force Phase II pumpability test program focused on the evaluation of six engine oils which exhibited significant yield stress or gelation index in slow-cool bench tests. The test oils were designed specifically to have abnormal flow properties and are considered to be non-commercial formulations. The light duty engines that primarily were used included the 2.2L I-4, 3.8L V-6, 4.0L I-6 and 4.6L V-8 engine, with some additional work in the 2.3L I-4, 1.9L I-4 and 3.0L V-6. Four laboratories contributed work, and each used different engines, and often, different cooling profiles, in attempts to better establish the importance of yield stress and gelation index to air-binding failures in modern engines.

For the majority of Phase II oils tested, air-binding failures were not detected. No pumping failures were detected with the phase II test oil LTEP 23, which had a gelation index of 16. Using the 4.6L V8 engine, an air-binding failure was detected only with LTEP 27, an oil that had caused air-binding failures in other work. The 2.2L I-4 engine did not fail by air-binding, but the 3.8L V-6 and 4.0L I-6 engines, which generally exhibit

[1] Consultant, Infineum, USA LP, 11735 Normont Dr., Houston, TX 77070.
[2] Project Leader, Consumers Union, 101 Truman Avenue, Yonkers, NY 10703-1057.
[3] HD Formulation Science Leader, Infineum USA LP, P.O. Box 536, Linden, NJ 07036.
[4] Vice President of Technology, Cannon Instrument Co., P.O. Box 16, State College, PA 16804-0016.
[5] Senior Research Associate, Imperial Oil, Research Dept., P.O. Box 3022, Sarnia, Ont., Canada N7T 8C8.
[6] Staff Research Engineer, General Motors Research Laboratories, P.O. Box 9055, Warren, MI 48090-9055.
[7] Senior Statistician, Infineum USA LP, P.O. Box 536, Linden, NJ 07036.

flow-limited failures, were "tricked" into producing air-binding failures by use of a reduced volume of test oil LTEP 28. This oil exhibited a gelation index of 40; it also exhibited a substantial yield stress (70-105 Pa) and a relatively low pumping viscosity when the engine pumping tests were run.

Keywords: yield stress, gelation index, pumping viscosity, air-binding failure, flow-limited failure, pumpability, light duty engines

Introduction

The ASTM Low Temperature Engine Performance Task Force Phase I engine pumpability studies were performed with a relatively fast cooling profile similar to that in the ASTM Standard Test Method for Predicting the Borderline Pumping Temperature of Engine Oil (D 3829). This fast cooling profile tends to supercool the oil and may not allow time for the complete formation and growth of wax crystals which can form networks responsible for significant yield stresses and/or higher viscosities. Also, the Phase I test oils were commercial oils, which were not purposely designed to exhibit gelation and yield stress characteristics.

The Phase II pumpability testing program's objective was to evaluate the relationship of yield stress and gelation index to the low-temperature pumping characteristics of modern engines. For this effort to succeed, it was necessary to find test oils with appropriate low temperature yield stress or gelation index values in bench tests, and then subject those oils to appropriate cooling profiles in engines, so that pumping characteristics could be monitored when the oil was in a gelled or highly viscous state.

The two major bench tests presently used to evaluate low temperature pumpability in the laboratory are the Mini-Rotary Viscometer (MRV) and the Scanning Brookfield Viscometer (SBT). While both the MRV and SBT can be operated at an almost infinite variety of cooling profiles, at the present time, only two cooling profiles are routinely used for the MRV (D 3829 or the ASTM method for Determination of Yield Stress and Apparent Viscosity of Engine Oils at Low Temperature (D 4684)), and only one profile for the SBT (Low Temperature, Low Shear Rate, Viscosity/Temperature Dependence of Lubricating Oils Using a Temperature-Scanning Technique (D 5133)). These profiles are described in the appropriate ASTM method and are graphically presented in Figure 1. The slow cooling profiles of the SBT and MRV TP-1 (D 4684) were developed, in part, to overcome the shortcoming of the D 3829 MRV test. The strength of the oil's network structure is indicated in the MRV by yield stress, and in the SBT by gelation index.

The oils selected for use in the Phase II program were designed specifically to have abnormal flow properties and are generally considered to be non-commercial formulations.

Four sponsors participated in the Phase II program. The engines included were the 2.2L I-4, 3.8L V-6, 4.0L I-6 and 4.6L V-8, plus some limited testing in the 2.3L I-4, 1.9L I-4 and 3.0L V-6 engines. Sponsors used a variety of cooling programs and independently selected oils to be tested from six Phase II test oils (LTEP 22-24, LTEP 26-28), as well as an occasional Phase I fluid (LTEP 2 and LTEP 3 were used). The objective here was to find a cooling profile that would cause a failure, and a variety of approaches were tried. Table 1 provides a summary of the laboratories, the oils evaluated, the cooling profiles applied, and the engines utilized for testing.

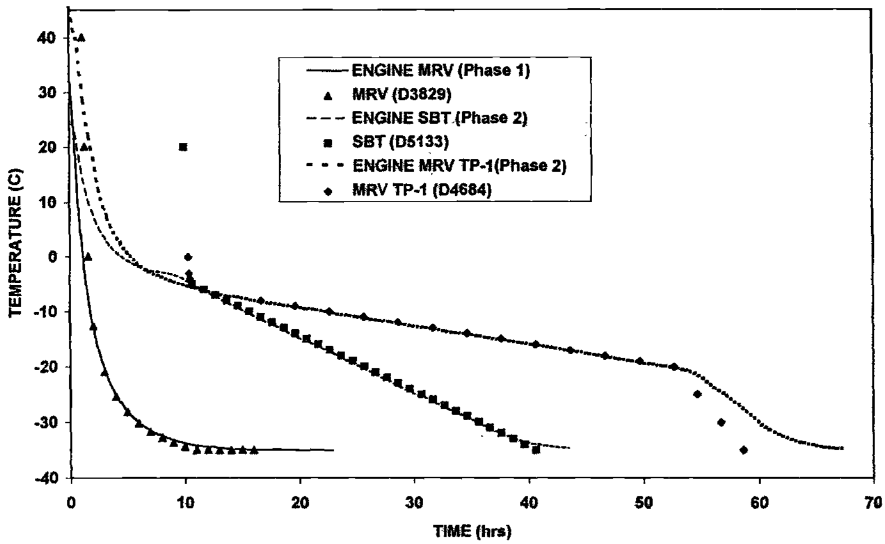


Figure 1 – *Comparison of Cooling Profiles, Standard Lab Tests and Those Used in Phase II Testing of 2.2L I4 Engine*

Table 1 - *Phase II Test Summary*

Sponsor	LTEP Oils	Cooling Program	Engine
Castrol	2, 22-24, 26	Modified Sioux Falls	4.6L V8
Castrol	2, 22-24,26	Alternate Sioux Falls	4.6L V8
Castrol	24	Slow cool rates 0.33 and 0.5 C/hour	4.6L V8
Castrol	27	Alternate Sioux Falls	4.6L V8
Texaco	24	MRV TP-1 and Scanning Brookfield	2.2L I4
Texaco	26	MRV TP-1	2.2L I4
Shell	3,28	Modified MRV TP-1	3.8L V6
Shell	3,28	Modified MRV TP-1	4.0L I6
Imperial Oil	23	Overnight, Alternate and Modified Sioux Falls	1.9L I4, 2.3L I4, 3.0L V6

4.6L V8 Engine Tests

Two cooling programs were targeted for this portion of the project. They are referred to as the Modified Sioux Falls and Alternate Sioux Falls cooling profiles. These cooling programs are derived from the natural overnight cooling that occurred in Sioux Falls during field pumpability failures in the winter of 1980-81 [*1-3*]. The Modified Sioux Falls profile rapidly cools the sump to an intermediate temperature and holds for a prescribed period of time. After this, the program continues with relatively rapid cooling until the final test temperature is reached and held for a defined period of time before the test. An example of the application of the Modified Sioux Falls cooling profile is shown in Figure 2 for an end-of-test target temperature of -34°C. The temperature curves given in Figure 2 are: the cell air temperature, the sump oil temperature at a location near the center above the pick-up screen, and the sump oil temperature adjacent to the pick-up screen. The cell air temperature was programmed to yield the required sump temperatures, as the offset between air temperature and sump temperature is nearly constant over the range of this program's test temperatures.

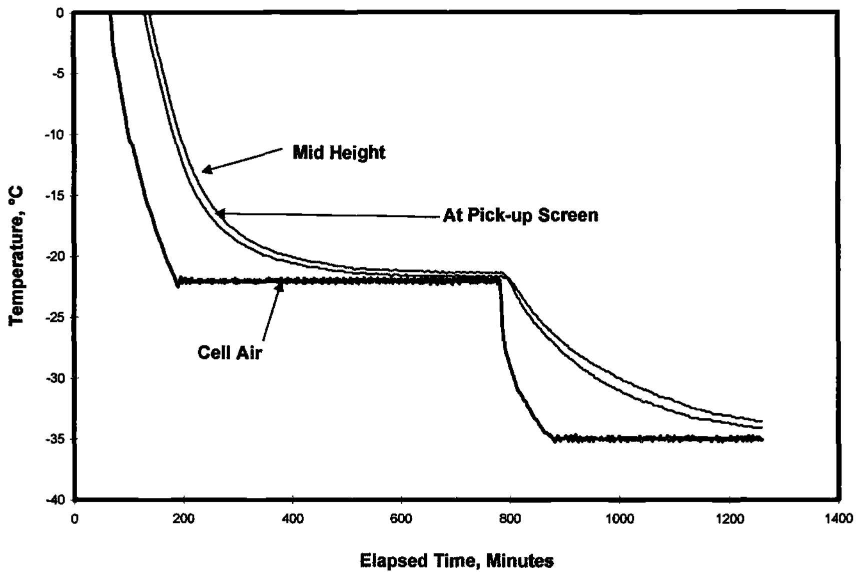


Figure 2 - *Modified Sioux Falls Cycle: Comparison of Cell to Sump Temperature*

A different cooling profile, called the Alternate Sioux Falls profile, is displayed in Figure 3. This profile rapidly cools the sump to an intermediate temperature, then slows the cooling rate to 0.5°C per hour over a prescribed temperature range, followed by relatively rapid cooling to the final test temperature. This type of profile was specifically suggested by the SBT developer as a way of promoting gelation by slowly cooling through the gelation index temperature. Again, the temperature curves for the three locations are shown in the figure. In this cooling program, sump temperatures nearly reach the ambient (soak) temperature about half-way through the programmed soak-time period.

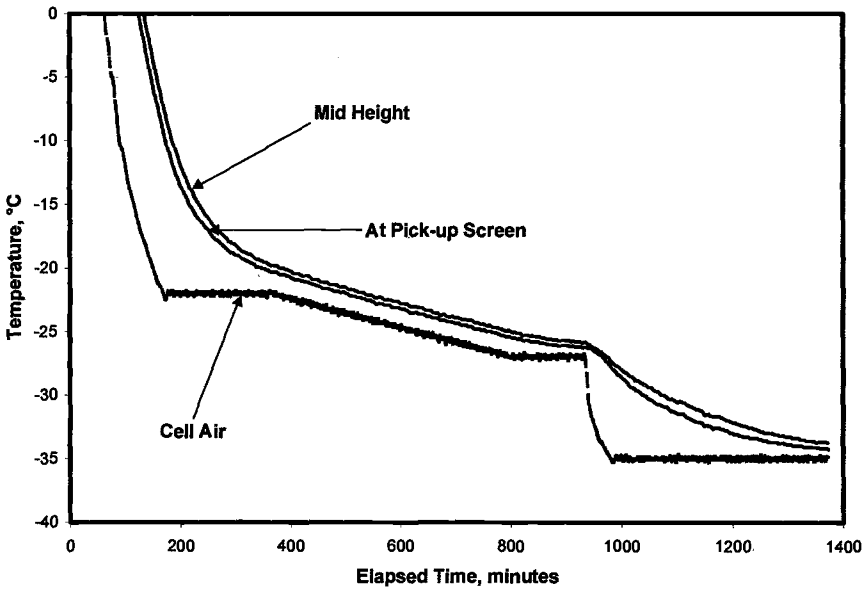


Figure 3 - *Alternate Sioux Falls Cycle: Comparison of Cell to Sump Temperature*

As a part of this program's effort to evaluate oils which exhibit significant yield stress (greater than 35 Pa) or gelation index, pumpability tests were conducted in the 4.8L V8 engine with LTEP oils 2, 22, 23, 24 and 26. (LTEP 2 is a commercial SAE 5W-30 used extensively in the Phase I pumpability program.) The oils were subjected to both the Modified and Alternate Sioux Falls cooling profiles before the motorized engine pumpability tests were initiated.

Oils 2, 23 and 24 were evaluated at 3°C below their minimum starting temperature, while oil 22 and 26 were evaluated at 6°C below their minimum start temperature. Note that the determination of the minimum starting temperature is reported in another paper in this series [*6*]. The pressurization time data are given in Table 2.

LTEP 2, 23 and 24 are SAE 5W-30 oils. They yield very similar pressurization times regardless of the cooling profile applied. LTEP 22 and 26 are SAE 10W-30 oils, and yield pressurization times very similar to the 5W-30 oils even though the evaluation temperature is six degrees below the minimum start temperature for 10W-30 oils. None of these five oils showed any evidence of air-binding during the tests. Air-binding behavior is demonstrated when the lubricant forms a gel-like structure with sufficient strength to result in the oil pump sucking a hole in the sump lubricant charge. Sporadic pressure spikes on the outlet side of the pump result as traces of oil are expelled into the oil galleys.

Table 2 - *Pressurization Data for 4.6L V8 under Rapid and Slow-Cool Cycles, LTEP 2, 22-24, 26*

Oil Code LTEP	Test Temperature °C	Pump Out, Time to 150 kPa, sec			Near Galley, Time to 10 kPa, sec		
		Rapid Overnight	Modified Sioux Falls	Alternate Sioux Falls	Rapid Overnight	Modified Sioux Falls	Alternate Sioux Falls
2	-34	3.7	5.3	3.3	20.8	24	24
23	-34	4.2	4.7	5.4	20.0	24	20
24	-34	4.9	4.1	3.5	15.5	21	14.5
22	-33	5.0	5.8	4.7	19.5	26.5	24
26	-33	3.5	4.3	3.9	17.5	13.5	30.5

To assess the effect of continuing the slow cooling portion to the final test temperature, two additional tests were run on LTEP 24. Using the Alternate Sioux Falls program as a starting point, the cooling profile was modified so that the engine/oil combination was exposed to a cooling rate of 0.33°C or 0.5°C per hour to the final test temperature of -34°C. The results of these tests are compared with the data from the normal Alternate Sioux Falls cooling result in Table 3. Extending the slow cool portion of the program at 0.33°C per hour to the final test temperature of -34°C did increase the pressurization time by a factor of two for this test. But neither of these extended slow cool tests showed any indication of air-binding.

Table 3 - *Pressurization Data for LTEP 24, Normal versus Extended Alternate Sioux Falls Cooling Cycles to -34°C*

		Pump Out, Time to 150 kPa, sec.			Near Galley, Time to 10 kPa, sec.		
Oil Code LTEP	Test Temperature °C	Std. Alternate Sioux Falls	Extended Slowcool -0.5 °C per hr	Extended Slowcool -0.33°C per hr	Std. Alternate Sioux Falls	Extended Slowcool -0.5°C per hr	Extended Slowcool -0.33°C per hr
24	-34	3.5	4	7.2	14.5	21.5	26

After the above results were obtained the question arose as to whether air-binding oils could be detected by this protocol or if the procedure was flawed. To help answer this question, LTEP 27 was added to the test program, for this oil had a high yield stress (by ASTM D 4684) in several labs at -20 and -25°C. This oil was reported previously in two SAE publications to produce air-binding failures under slow-cool conditions [*7*, *8*]. The SAE 10W-30 oil was therefore exposed to the Alternate Sioux Falls cooling program in the 4.6L V8 engine to a final test temperature of -33°C. Under these conditions, no pressures were obtained at any location in the engine before 60 seconds had elapsed. This was somewhat surprising until it was recognized that the previous studies with this oil had been conducted at temperatures of about -25°C, and that given the viscosity-temperature characteristics of this oil, it probably had been rock solid at -33°C. The test therefore was repeated with a fresh oil charge, this time targeting a -25°C sump temperature. The results, given in Figure 4, show a classic air-binding failure, as seen previously with this oil. This test demonstrates that the test protocol can produce air-binding failures if the oil exhibits a high yield stress condition at the test temperature.

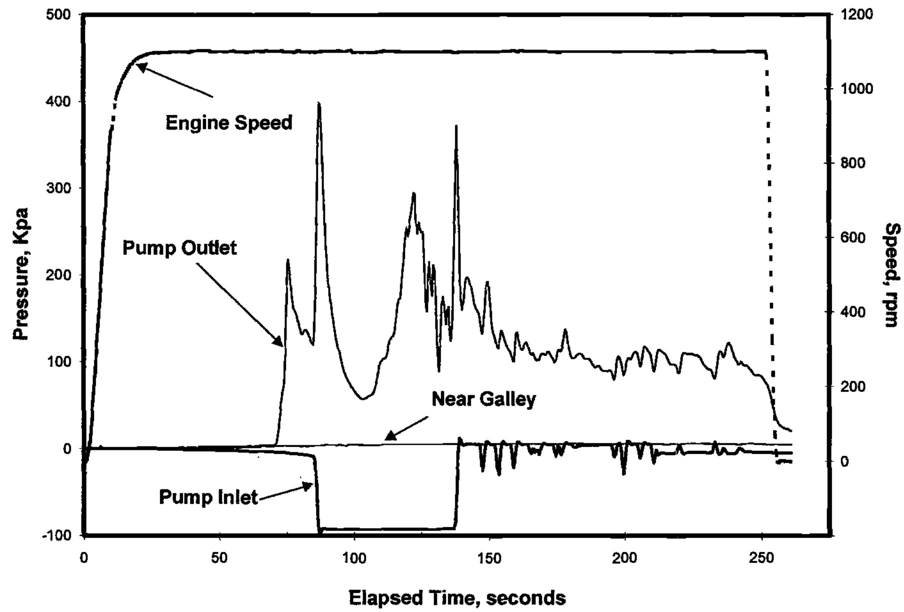


Figure 4 - *LTEP 27 in 4.6L V8 Engine: Alternate Sioux Falls Cooling Program to -25°C*

2.2L I4 Engine Tests

The experimental goal in this portion of the program was to try to reproduce the SBT and MRV TP-1 profiles in the cold room, and to test some of the Phase II series oils using these profiles. The engine pumpability results were then evaluated relative to the laboratory results obtained with the various viscosity techniques. LTEP 24 and LTEP 26 were selected for study mainly because of their rheological characteristics at -35°C, but they are also representative of the two most common winter viscosity grades (SAE 5W-30 and 10W-30). The -35°C temperature was chosen for several reasons. This is the lowest temperature at which rheological data could be obtained with the MRV equipment in-hand. Those data indicated that cooling cycle had a significant impact on the flow properties of the oil (Table 4). Since both of these oils easily met the pumpability criteria in Phase I with the fast overnight (MRV) cooling cycle, this program would determine how the change in the rheological properties would affect the pumpability of the oils.

Table 4 - *Rheological Properties of Oils Used in Phase II Pumpability Testing with 2.2L I4 Engine**

	D 3829 MRV		D 4684 MRV TP-1		D 5133 SBT **	
Test Oil (LTEP)	Viscosity, mPa·s	Yield Stress, Pa	Viscosity, mPa· s	Yield Stress, Pa	Gel Index	Gelation Temp., °C
24	30,800	< 35	48,000	105	33.1	-20
26	51,200	< 35	86,000	105	28.1	-11

* All viscosity and yield stress results at -35°C

** Viscosity by SBT at -35°C was >40,000 mPa·s for both oils.

The cold room could not achieve the initial rapid cool down to -5°C of the laboratory SBT and MRV techniques, shown in Figure 1. However, it was able to match the slow cooling profile of these methods through the -5 to -35°C temperature region believed to be critical for wax formation. At the end of the cooling cycles, the engine was held at the final temperature for an additional 3 hours to assure temperature uniformity throughout the oil prior to initiating the pumpability test.

The pressure traces for each oil with each cooling cycle, including the overnight profiles from Phase I testing, are shown in Figures 5 through 9. In general, the pressure profiles are very similar except those from LTEP 26 with the MRV TP-1 cycle. As per the protocol, this test was stopped because no pressure was detected at the near galley after 60 seconds. The rest of the tests were completed successfully. Slowing the cooling rate of the oil (i.e. going from the fast overnight cooling to the SBT cycle and then to the MRV TP-1 cycle) extended the time to initial pressurization at all locations downstream of the pump.

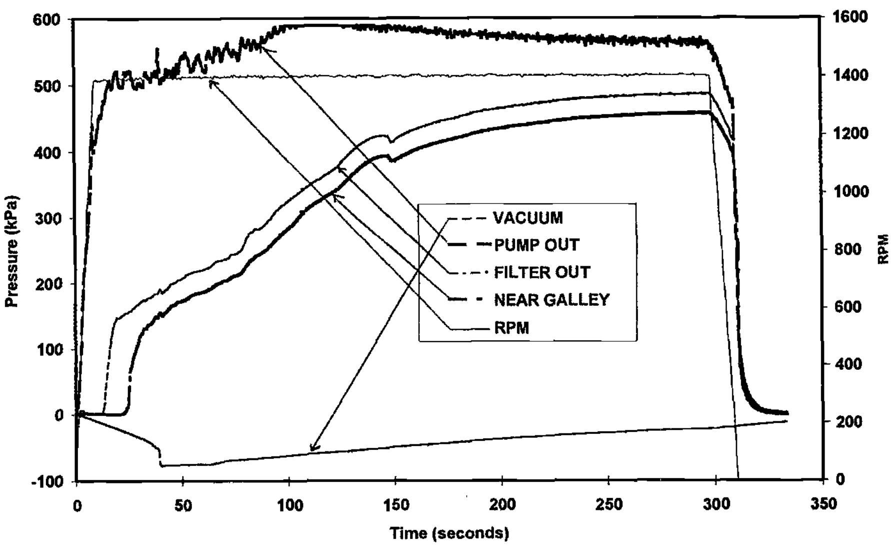


Figure 5 – *Oil Pressure Traces, LTEP 24, Overnight Cooling, 2.2L I4, -35°C*

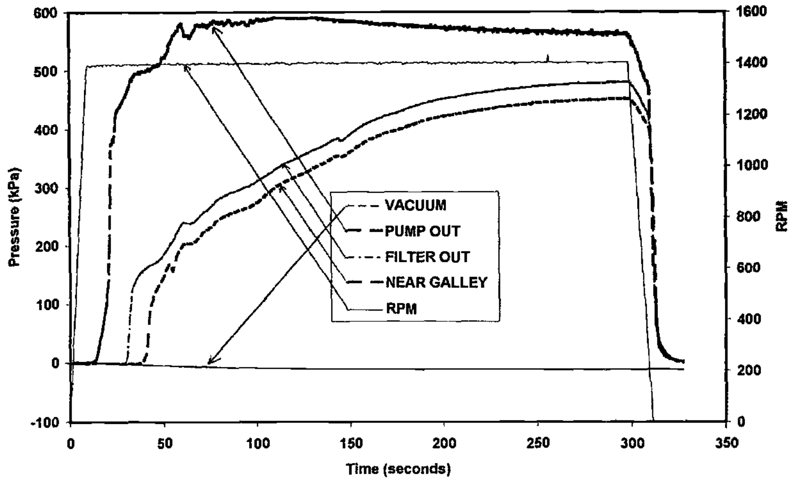


Figure 6 – *Oil Pressure Traces, LTEP 24, SBT Cooling, 2.2L I4, -35°C*

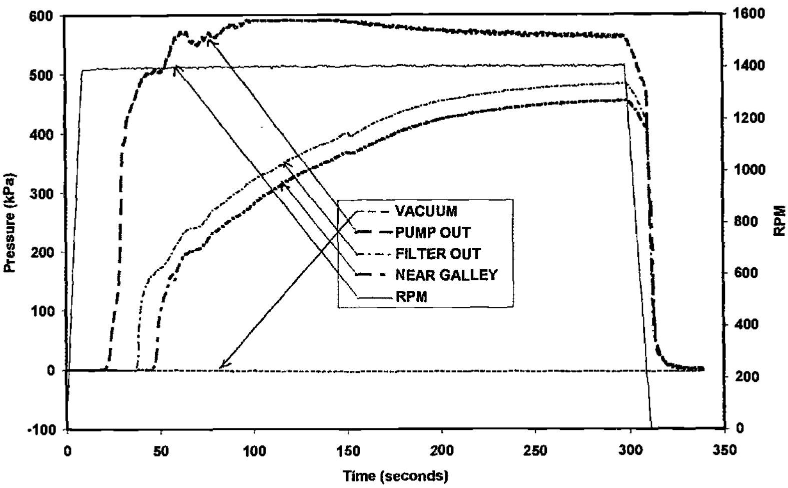


Figure 7 – *Oil Pressure Traces, LTEP 24, MRV TP-1 Cooling, 2.2L I4, -35°C*

Vacuum at the pump inlet with the slower cooling cycles was almost non-existent suggesting that the oil in the sidearm connecting the oil pick-up tube to the transducer is so viscous that the normal force generated by the oil passing through the pick-up tube is not enough to cause it to exert pressure on the sensor. Despite the significant yield stresses demonstrated by these oils with the MRV TP-1 cycle in the lab, there was no indication of air-binding in any of these pumpability results.

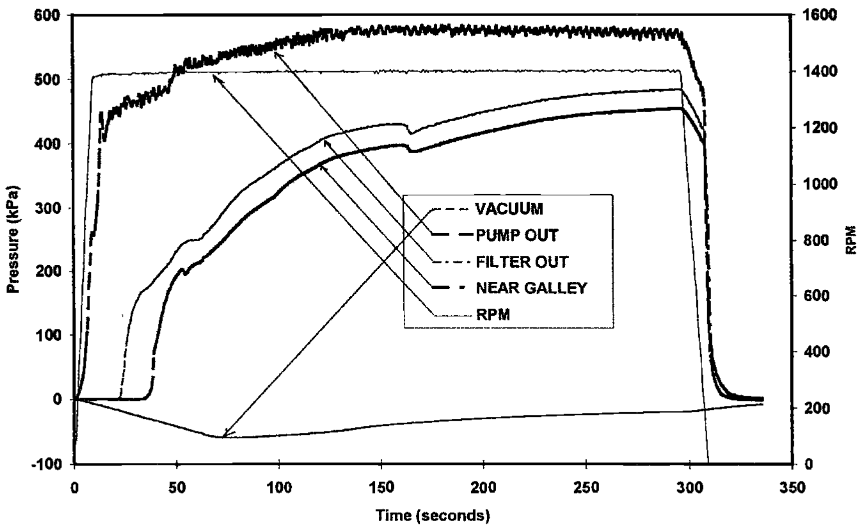


Figure 8 – *Oil Pressure Traces, LTEP 26, Overnight Cooling, 2.2L I4, -35°C*

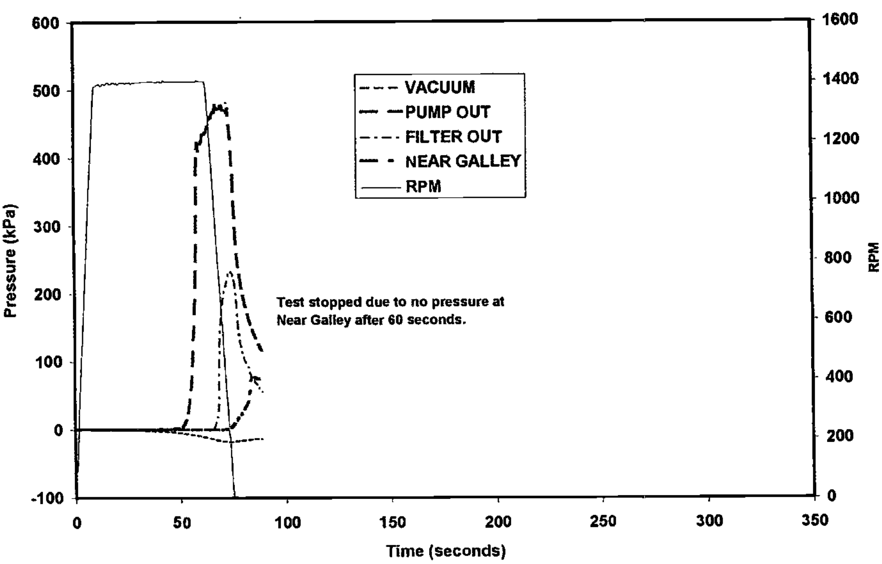


Figure 9 – *Oil Pressure Traces, LTEP 26, MRV TP-1 Cooling, 2.2L I4, -35°C*

Figures 10 and 11 show the time required for these oils to reach the flow limited pumpability criteria after the pump and at the near galley locations under the different cooling cycles. Again, there is no data point for LTEP 26 with the Scanning Brookfield cycle because this test was not performed. As would be expected from its overall higher viscosities, LTEP 26 requires more time to reach the pumpability criteria than LTEP 24.

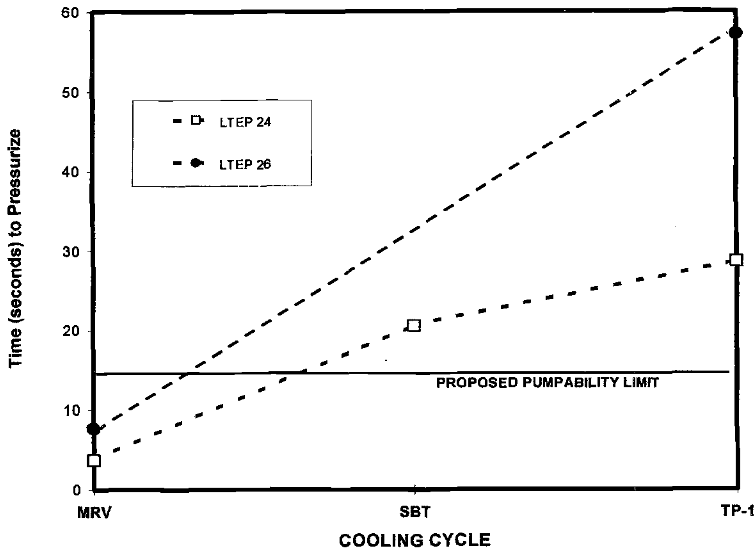


Figure 10 – *Time to 150 kPa at Pump Out with Various Cooling Cycles, 2.2L I4, -35°C*

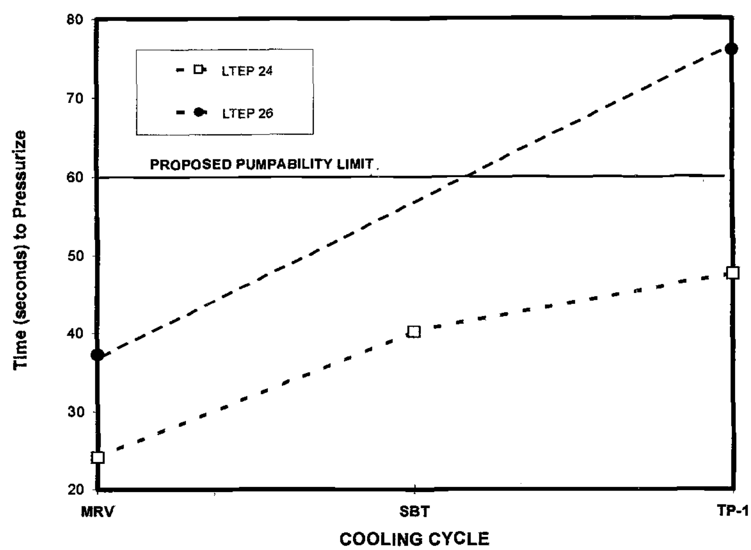


Figure 11 – *Time to 10 kPa at Near Galley with Various Cooling Cycles, 2.2L I4, -35°C*

The difference in time between the two oils is larger with the TP-1 (D 4684) cycle than with the D 3829 cycle. The results also show that both oils met the proposed Pump Out criterion only with the D 3829 cooling cycle, while it appears that both meet the near galley criterion with both the MRV and SBT cycles. With the TP-1 cooling cycle, only LTEP 24 meets the near galley criterion for pressurization. Contrary to the Phase I testing, these results suggest that the Pump Out criterion is more difficult to meet than that at the near galley.

It would be difficult, if not impossible, to generate an overall correlation between the pumpability limits and oil viscosity across all of the cooling cycles because of the variability in the MRV TP-1 viscosity results caused by the presence of a wax network in the oil. In addition, the oil viscosities at -35°C are beyond the capability of the SBT technique. However, the results indicate that to meet the Pump Out criteria, the MRV viscosity can be higher than 50,000 mPa·s with this engine if the oil shows no significant yield stress. At the near galley, the limiting viscosity is between 50,000 and 85,000 mPa·s even with an oil having a significant yield stress. Overall, these results support the MRV viscosity limits developed during Phase I testing. It also appears that the MRV technique has the best potential for predicting flow limited pumpability failures in engines. In order for the SBT to become a useful tool for predicting flow limited pumpability failures, it would need to be modified to measure higher viscosities or shear stresses.

Since air-binding failure was not detected in any of the tests with this engine, it is impossible to come to definitive conclusions as to whether MRV yield stress and/or SBT Gel Index are good indicators of an oil's tendency to air bind. However, these results do suggest that, if these techniques are to be used for this application, the yield stress and Gel Index limits should be higher than those demonstrated by LTEP 24 and 26. It should be noted that this statement is valid for this engine and for the cooling cycles shown in Fig. 1 and may not be the case if alternate cooling cycles are employed.

3.8L V6 and 4.0L I6 Engine Tests

The objective of this Phase II pumpability testing was to determine whether either of the engines, which experienced flow-limited failures in the Phase I pumpability program, would undergo an air-binding failure with a test oil that had a significant yield stress, i.e. about 105 Pa, combined with a relatively low viscosity. The near galley calculated D 3829 critical viscosities ranged from 31 to 40 Pa(s) at average minimum pumping temperatures from the Phase I (overnight) program using the more critical 4.0L engine. Thus, the test oil target MRV viscosity needed to be less than this, in order to avoid flow-limited failures in this engine.

Alternate Cooling Profile MRV Tests

It was anticipated that a more extended cooling profile than D 3829, such as that used for the TP-1 MRV test (ASTM D 4684) would be more likely to subject oils to conditions where yield stress would develop. MRV yield stress and viscosity often depend upon the cooling profile that is applied. To identify an oil with the target properties, an MRV apparatus was fitted with a specially-designed computer control system so that LTEP test oils could be exposed to a variety of extended temperature profiles. Once a discrete temperature profile was found that produced the targeted yield stress and viscosity, this proxfile would be applied to the oil in cold room engine test pumping studies.

There are three discrete cooling steps of the TP-1 MRV profile. For the TP-1 MRV test at -25°C, after initial heating to 80°C, the oil is shock cooled to -5°C. This step will be referred to as Step 0. This is followed by a short cooling period during which the oil temperature drops to -8°C (Step 1). Then a linear cooling period is imposed where temperature is reduced to -20°C at the rate of 0.33 degrees per hour (Step 2). This is followed by a more rapid cooling stage of 2.5 degrees per hour until the final test temperature is reached (Step 3). The apparatus is then held at this temperature for ½ hour before yield stress and viscosity determinations are made. Steps 1-3 are listed in Table 5.

All of the alternative cooling profiles listed in Table 5 had the same Step 0 as the TP-1 profile; the time for heating to 80°C and shock cooling to -5°C are identical in all profiles. All of the *time sequences* required for Steps 1 and 2 of the alternative cooling profiles also were the same as the TP-1 profile. For example, the time required to complete Step 1 of the TP-1 MRV was equivalent to any of the alternative profiles, and similarly for Step 2. However, the cooling rates for Steps 1 and 2 differed between TP-1 and the alternative profiles. Table 5 identifies Steps 1 and 2 in terms of the temperature achieved at the end of the time period for the step, and in the case of Step 3, the measurement temperature. Thus, each Step 1 begins at -5°C (the temperature at the end of Stage 0), and ends at the temperature shown in the Step 1 column. Each Step 2 begins at the temperature shown for Step 1 and ends at the temperature given in the Step 2 column. The third stage of TP-1 always involves cooling to the measurement temperature at a rate of 2.5°C per hour to the test temperature, followed by the measurement of yield stress and viscosity after ½ hour at the measurement temperature. If there was no decrease in temperature from the end of Stage 2 and the measurement temperature, as in the profile TP-13(-25)-25, then the total time for Stage 3 was equivalent to that of the TP-1 profile when the final test temperature is also -25°C. The third stage time period for the alternate profiles exceeded that of the TP-1 profile for a -25°C test if the measurement temperature was lower than -25°C.

Yield stress observations, combined with relatively low viscosities, were observed for LTEP 22, 24 and 26. However, none of the oils produced 40 gram-to-turn results that would be indicative of a 105 Pa yield stress failure. LTEP 28 was the last Phase II oil to be made available for testing and appeared to be most suitable for air-binding evaluation on the basis of TP-1 MRV testing[1]. Alternative cooling profiles were not run on this oil, for there was no need to find a more suitable cooling profile than TP-1. In the TP-1 MRV test (D 4684), yield stresses were in the 105 Pa range and viscosities were below 60,000 mPa·s. This oil was selected for evaluation in both the 3.8L V6 and 4.0L I6 engines.

Engine Pumpability Tests with LTEP 28

[1] Because this lab conducted Phase II testing after the other labs had essentially completed their programs, LTEP 28 testing was only conducted in the 3.8L V6 and 4.0L I6 engines.

Table 5 - *Variable Temperature MRV Experiments - LTEP Oils*

		Variable Temperature MRV Results, Including TP-1 Profile					
			Test Temperatures for end of each cooling stage, °C				
LTEP No.	SAE Grade	Profile ID	Step 1 End	Step 2 End	Step 3 End	MRV Viscosity, mPa·s Avg. (Y. Str., Pa)	No. of Tests
1	0W-30	TP-1 @ -35	-8	-20	-35	---	---
1	"	TP-13(-25)-25	-13	-25	-25	2,630 (<35)	3
1	"	TP-18(-30)-30	-18	-30	-30	4,100 (<35)	1
1	"	TP-23(-30)-30	-23	-30	-30	4,200 (<35)	1
1	"	TP-23(-35)-35	-23	-35	-35	8,042 (<35)	1
2	5W-30	TP-1 @ -30	-8	-20	-30	---	---
2	"	TP-13(-25)-25	-13	-25	-25	4,000 (<35)	2
2	"	TP-18(-30)-30	-18	-30	-30	8,500 (<35)	1
2	"	TP-23(-30)-30	-23	-30	-30	8,400 (<35)	1
2	"	TP-23(-35)-35	-23	-35	-35	20,900 (<35)	1
3	10W-30	TP-1 @ -25	-8	-20	-25	---	---
3	"	TP-13(-25)-25	-13	-25	-25	11,300 (<35)	2
3	"	TP-18(-30)-30	-18	-30	-30	29,000 (<35)	1
3	"	TP-23(-30)-30	-23	-30	-30	25,000 (<35)	1
3	"	TP-23(-35)-35	-23	-35	-35	106,100 (<35)	1
22	10W-30	TP-1 @ -25*	-8	-20	-25	8,500 (<35)	1
22	"	TP-13(-25)-25	-13	-25	-25	10,300 (35**)	9**
22	"	TP-13(-25)-25	-13	-25	-25	10,500 (70)	2
22	"	TP-18(-30)-30	-18	-30	-30	22,600 (35)	1
22	"	TP-23(-30)-30	-23	-30	-30	20,100 (<35)	1
22	"	TP-23(-35)-35	-23	-35	-35	56,200 (<35)	1
23	5W-30	TP-1 @ -30*	-8	-20	-30	11,900 (<35)	1
23	"	TP-13(-25)-25	-13	-25	-25	4,300 (<35)	2
23	"	TP-18(-30)-30	-18	-30	-30	10,300 (<35)	2
23	"	TP-23(-30)-30	-23	-30	-30	10,100 (<35)	2
23	"	TP-23(-35)-35	-23	-35	-35	24,900 (<35)	1
24	"5W-30"	TP-1 @ -30*	-8	-20	-30	14,500 (35)	2
24	"	TP-13(-25)-25	-13	-25	-25	7,440 (70)	2
24	"	TP-18(-30)-30	-18	-30	-30	17,900 (35)	1
24	"	TP-23(-30)-30	-23	-30	-30	14,000 (<35)	1
24	"	TP-23(-35)-35	-23	-35	-35	37,100 (<35)	1
26	10W-30	TP-1 @ -25*	-8	-20	-25	15,200 (70)	2
26	"	TP-13(-25)-25	-13	-25	-25	24,000 (35)	2
26	"	TP-18(-30)-30	-18	-30	-30	63,500 (35)	1
26	"	TP-23(-30)-30	-23	-30	-30	18,600 (<35)	1
26	"	TP-23(-35)-35	-23	-35	-35	44,300 (<35)	1
26	"	TP-1 @ -30	-8	-20	-30	37,700 (70)	2

* using variable temperature MRV apparatus for TP-1 measurement.

** of 9 measured samples, 5 had yield stress <35 Pa.

The test protocol used for evaluation of LTEP 28 was identical to that used for Phase I testing except that a different cooling protocol was employed, and the control temperature sensor for the cold room was inserted into the engine through the dipstick tube hole, in order for the crankcase oil to approach the closest approximation of the desired TP-1 temperature profile at -25°C.

The oil temperature drop in the engine sump lagged the oil temperature in the TP-1 MRV when there was a rapid drop in the temperature of the TP-1 profile. This occurs in the TP-1 profile after the initial heat stage, when the oil is shock-cooled, and during the last step in the profile, when the oil is cooled at a rate of 2.5°C per hour.

To minimize the difference in cooling rate during shock-cooling, the engine was cooled first to -5°C overnight, without oil in the sump. LTEP 28 or LTEP 3 was preheated to 80°C for two hours outside of the cold room, then immediately added to both the engine and to the MRV in the cold room. The cold room was maintained at -5°C for 30 minutes after oil addition, before the slow-cool profile was applied. A comparison of the TP-1 MRV cooling profile (without Stage 0) and the bulk oil temperature actually obtained for LTEP 28 in the crankcase of the 4.0L I6 engine on 5/03/96 is given in Figure 12. The engine cooling profile - indicated as "sump temperature" - in the figure was used for all tests on LTEP 28 and LTEP 3, except that the last cooling stage, below -20°C, was extended or shortened, depending on the targeted test temperature for the pumping evaluation.

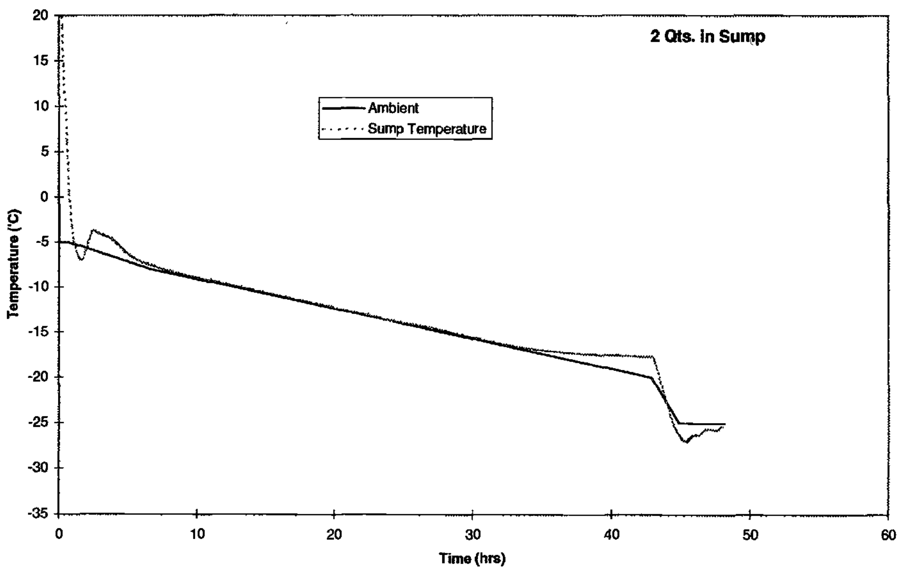


Figure 12 - *TP-1 MRV Type Cooling Profile (LTEP-28, 4.0L I6 engine)*

Side-by-side cold room MRV results were not obtained in every case. In one instance where a cold room MRV result was not obtained, the variable-temperature MRV apparatus in the laboratory was used to supplement cold room MRV results. This was accomplished by programming the MRV cooling cycle to duplicate the temperatures recorded in the sump during the cold soak. The cooling profile contained the heating stage of TP-1 (identified as Stage 0) and the substitution of "sump temperature" profile for the TP-1 Steps 1, 2, and 3.

The engine test conditions, test results, as well as MRV results using the 2-day cooling profile are summarized in Table 6. LTEP 28 was first tested using full charges of oil in both the 3.8L V6 and 4.0L I6 engines. At final test temperatures of between -25°C and -26°C, pressure rises were observed which were judged to be normal for both engines. There was no evidence of failure (see Figures 13 and 14). Nominal temperatures given are the target temperatures for the cold room at test time. The 3.8L V6 and 4.0L I6 test columns represent the measured oil sump temperature at the time of test.

The cold room MRV block temperatures were slightly colder than the engine sumps; measured viscosities were observed to be 20,700 mPa·s or less in each case with yield stresses that required either a 40 or 50 gram weight to turn the spindle of the MRV (105-140 Pa yield stress). Laboratory variable temperature MRV measurements at -25.0°C using the "sump temperature" profile provided slightly lower viscosities for LTEP 28, and between 70 and 105 Pa yield stress in repeat testing.

Reduction of oil sump charge to 3¼ quarts did not provide a pumping failure as judged from pressurization times at oil sump temperatures of about -23°C, but tests at -25°C using 2 quart sump volumes of LTEP 28 caused repeatable air-binding failures (Figures 15, 16) [*4*].

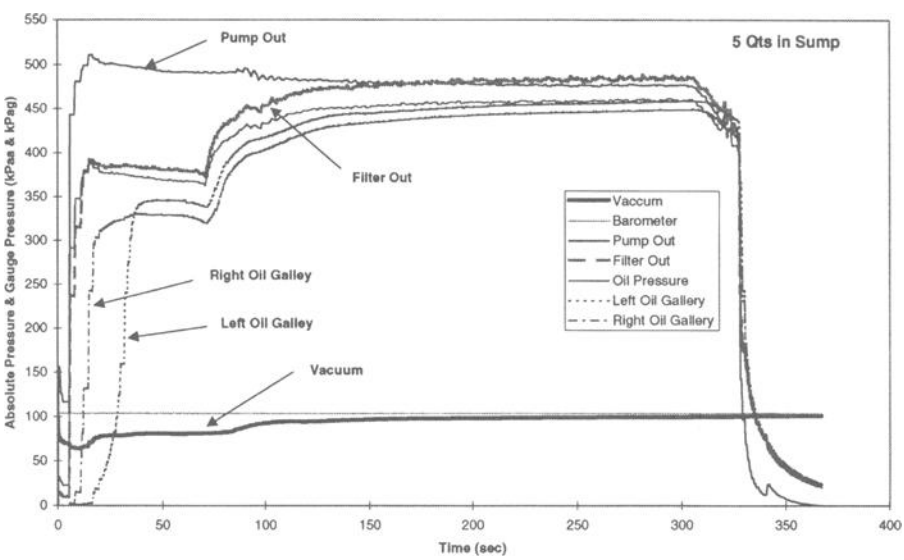


Figure 13 - *3.8L V6, 5 Qt. Oil Charge, LTEP-28, Slow Cooling to -25°C*

Table 6 - *Summary cf LTEP Task Force Phase II Pumpability Testing @ Shell Westhollow Technology Center: Air-Binding Work*

Fig. No.	Oil Sump Charge US Qts	Date	LTEP Oil	Nominal Test Temp (°C)	3.8L V6 Test Temp. (°C)	4.0L I6 Test Temp. (°C)	MRV Temp. (°C)	MRV Vis. mPa·s	MRV Y. Stress Pa	3.8L Motor Speed rpm	4.0L Motor Speed rpm	3.8L Pass?	4.0L Pass?
13	5	4/10/96	28	-25.0	-25.2	---	---	---	---	1157.5	---	Pass	---
14	6	4/17/86	28	-25.0	---	-25.4	-27.1	20,700	105	---	989.2	---	Pass
15	2	5/3/96	28	-25.0	---	-25.6	-26.3	18,300	140	---	994.0	---	**Fail**
16	2	5/9/96	28	-25.0	-25.1	---	-25.5	15,900	105	1165.8	---	**Fail**	---
---	3¼	5/22/96	28	-25.0	-23.3	---	---	---	---	1168.1	---	Pass	---
---	3¼	7/17/96	28	-25.0	-22.9	---	---	---	---	1174.8	---	Pass	---
---	2	8/7/96	28	-25.0	-25.0	---	-25.0*	12,500*	70*	1170.3	---	**Fail**	---
							-25.0*	12,300*	105*				
							-25.0*	11,600*	70*				
17	2	8/14/96	3	-25.0	-25.9	---	---	---	---	1169.7	---	Pass	---
18	2	9/12/96	3	-25.0	---	-26.0	---	---	---	---	1000.5	---	Pass

Notes:

* Result obtained from laboratory variable temperature MRV apparatus using cooling profile obtained in the Cold Room.
* Cooling profiles were reasonable approximations to the MRV TP-1 -25°C Profile.
* Flush Method: Engine flushed with test oil at room temperature, then drained; then 80°C oil flushed into engines & MRV after holding cold box at -5°C overnight.
* Engines motored with 60 hp DC motors.

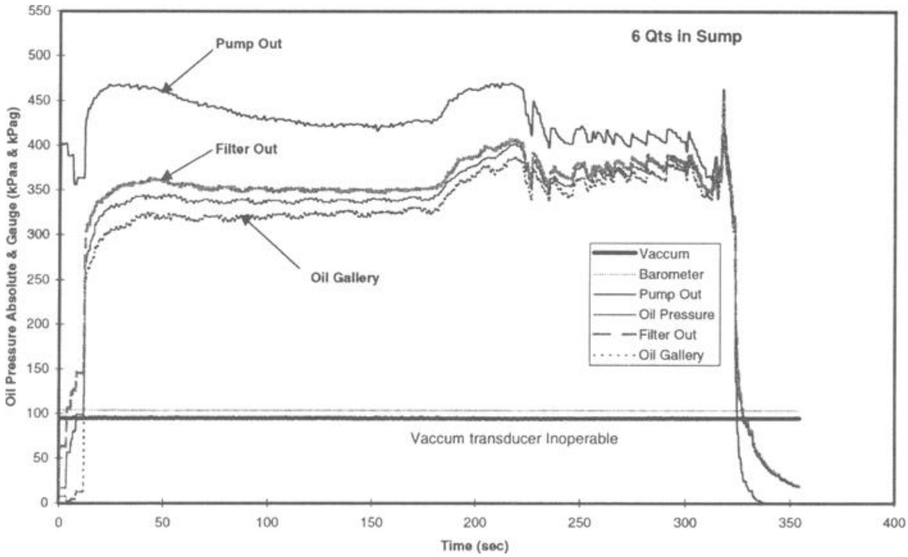


Figure 14 - *4.0L V6, 6 Qt. Oil Charge, LTEP-28, Slow Cooling to -25°C*

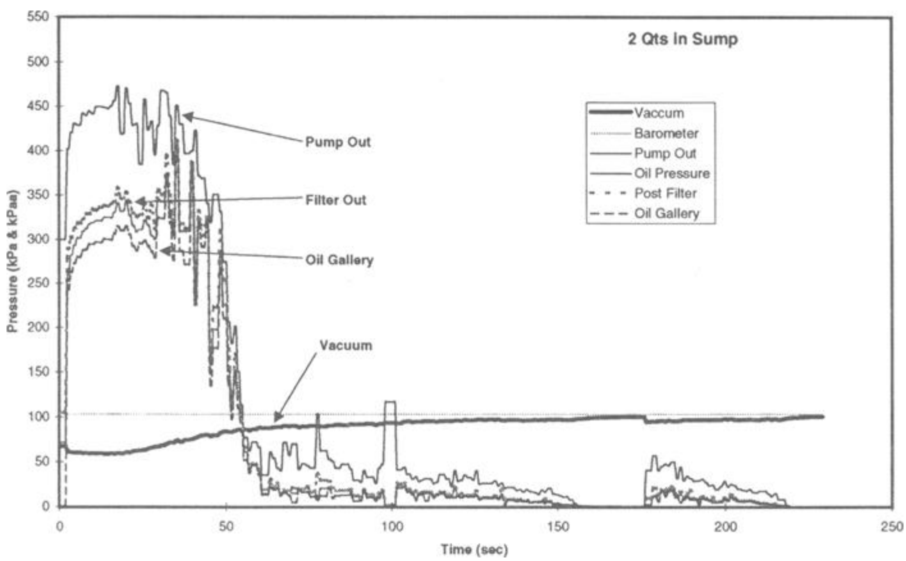


Figure 15 - *4.0L I6, 2 Qt. Oil Charge, LTEP-28, Slow Cooling to -25°C*

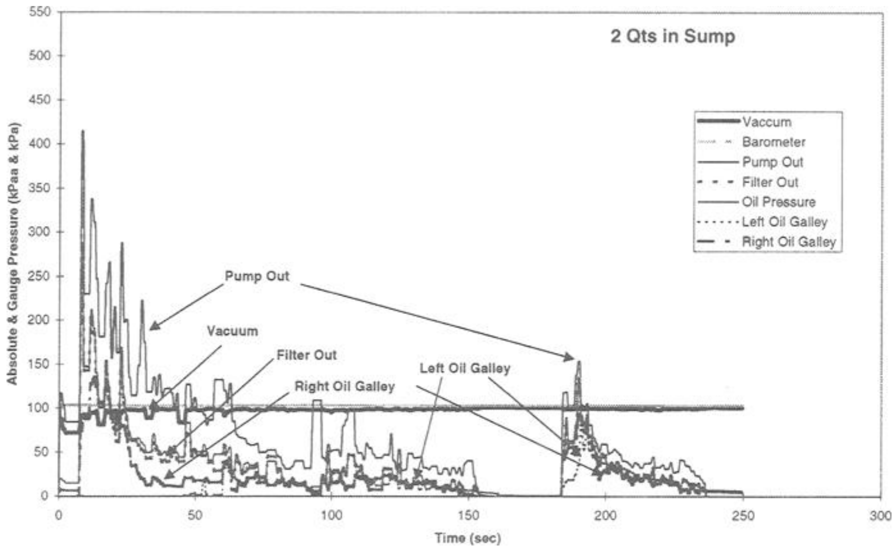


Figure 16 - *3.8L V6, 2 Qt. Oil Charge, LTEP-28, Slow Cooling to -25°C*

At room temperature, a quantity of 2 quarts of oil was determined to be adequate to provide sustained oil pressurization with LTEP 28 in these engines. However, further confirmation was needed to ensure that 2 quarts of oil sump charge would normally be sufficient to sustain crankcase oil volume height above the pump inlet screen, unless an oil gelling condition was present. LTEP 3 was therefore selected for low oil-volume testing. This oil had no demonstrable yield stress measurement at -25°C, and had only exhibited flow-limited failures at much lower temperatures. With only 2 quarts of each oil in the engines, no air-binding conditions reminiscent of LTEP 28 was observed in either engine at test temperatures of -26°C (Figures 17, 18). Yield stress was not observed for LTEP 3 when the oil was evaluated in the laboratory variable temperature MRV apparatus using the "bulk oil T" profile and with a measurement temperature of -25°C (these data are not shown in Table 6).

Since oil pick-up heights above the pan bottom were estimated to be 1.0 cm for the 4.0L engine and 0.5 cm for the 3.8L engine, air-binding failures must have occurred when oil height above the inlet was no more than 5.2 cm in the 4.0L engine and 5.8 cm in the 3.8L engine. The oil height above the bottom of the oil pans on the 3.8L V-6 and 4.0L I-6 engines were measured at full and partial volumes of oil (see Table 7).

Both the 3.8L V6 and 4.0L I6 engines undergo flow-limited pumping failures under full-sump conditions. By lowering oil sump volume to 2 quarts with LTEP 28, however, both engines were transformed into yield stress sensitive engines, and air-binding failures were observed. These failures occurred with an oil that, at the same time, exhibited 105 Pa yield stress in the cold room MRV. Failures did not materialize when LTEP 3 was tested under similar conditions. This oil, with relatively low TP-1 MRV viscosities at -25°C, and no observed yield stress, achieved full pressurization with only a 2 quart charge.

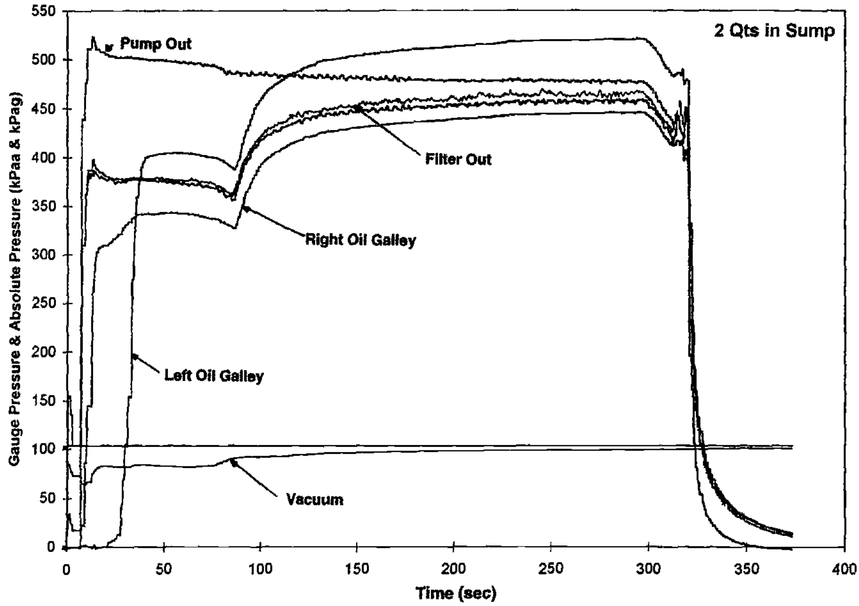


Figure 17 - *3.8L V6, 2 Qt. Oil Charge, LTEP-3, Slow Cooling to -25°C*

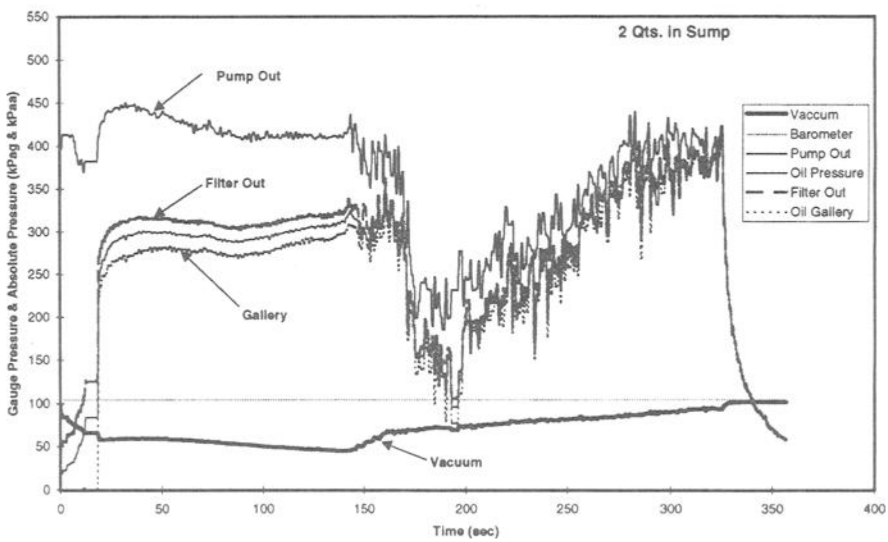


Figure 18 - *4.0L I6, 2 Qt. Oil Charge, LTEP-3, Slow Cooling to -25°C*

Table 7 - *Estimated Oil Heights in 4.0L I-6 and 3.8L V-6 Engines*[a]

Oil Volume US quarts	4.0L I-6 Oil Height, cm	3.8L V-6[b] Oil Height, cm
5	11.0	11.2
4	9.4	9.6
3	7.9	8.1
2	6.2	6.3

[a] Oil pick-up heights above pan bottom approx. 0.5 cm for 4.0L I-6 and 1.0 cm for 3.8L V-6.

[b] The 3.8L V-6 used in this study was Series 1 type (Manufacturer's designation). Oil Height data provided in the OEM survey (Table C-3, Appendix C) refers to Series 2 type design of this engine, with a slightly smaller oil height.

1.9L I4, 2.3L I4 and 3.0L V6 Engine Tests

The purpose of this study was to compare the pumpability characteristics of LTEP 23 (an SAE 5W-30 with good D 4684 MRV characteristics, but relatively high Gel Index (16 at -22.5°C)) under various cooling conditions, to determine whether gel index could be related to pumpability (in particular, air-binding tendencies). The engines were tested under 3 cooling conditions: (a) rapid overnight cooling (as in Phase I pumpability), (b) 24 hr. modified Sioux Falls cycle, with intermediate temperature of approximately -22.5°C as in Figure 19, and (c) a 48 hr alternate Sioux Falls cycle where the oil was slowly cool-

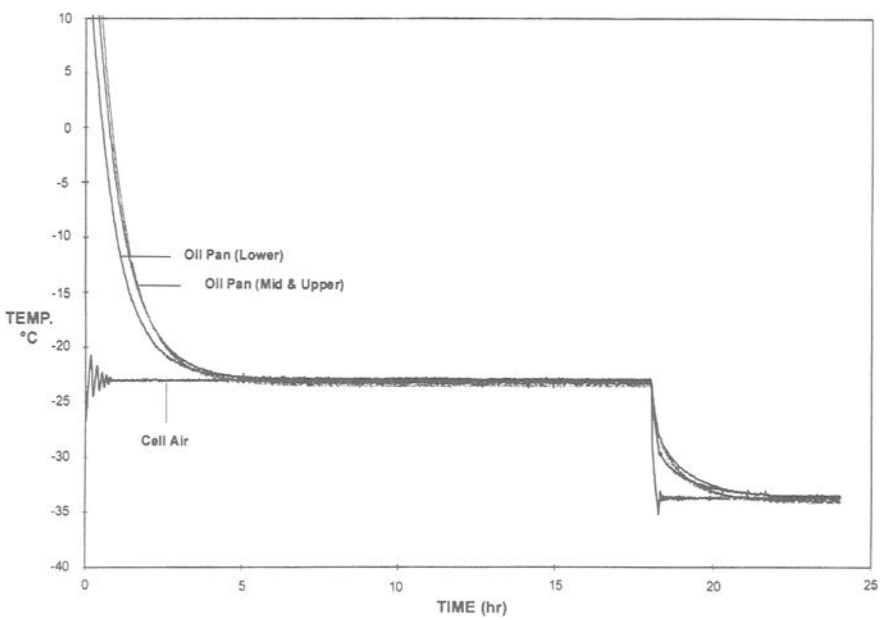


Figure 19 – *Modified Sioux Falls Profile, 2.3L I4 Engine, LTEP 23*

ed (0.33°C/hr) from -20 to -29°C (i.e. above the GI temperature, to several degrees below it, as recommended by the SBT developer) - see Figure 20. Final test temperature was approximately -33 to -35°C ambient (oil temperatures ~-33 to -34°C), which was slightly lower than the lowest minimum starting temperature of the three engines. The results of these tests are summarized in Table 8.

It is apparent that, for a given engine, the pumpability characteristics of LTEP 23 were very similar regardless of cooling cycle, with rapid pressurization observed after start-up. No evidence of air-binding failure was detected in any of these tests, and in no instance did the pumpability event approach the limiting criteria for Pump Out or Near Galley times used in this program (Nor did modified, variable temperature MRV experiments detect a yield stress with this oil; see Table 5). The results for the 1.9L I4 engine should be especially noted, since this engine has the lowest oil height and a small L/D^4 ratio, and hence, was considered to be particularly susceptible to air-binding failure.

To put these pressurization times into perspective, Table 9 lists the pumpability data generated on LTEP 2, also an SAE 5W-30 grade (and viscometrically well-behaved), at approximately the same oil temperatures, after overnight rapid cooling. It is apparent that these data are in line with the results generated with LTEP 23 under the various cooling conditions.

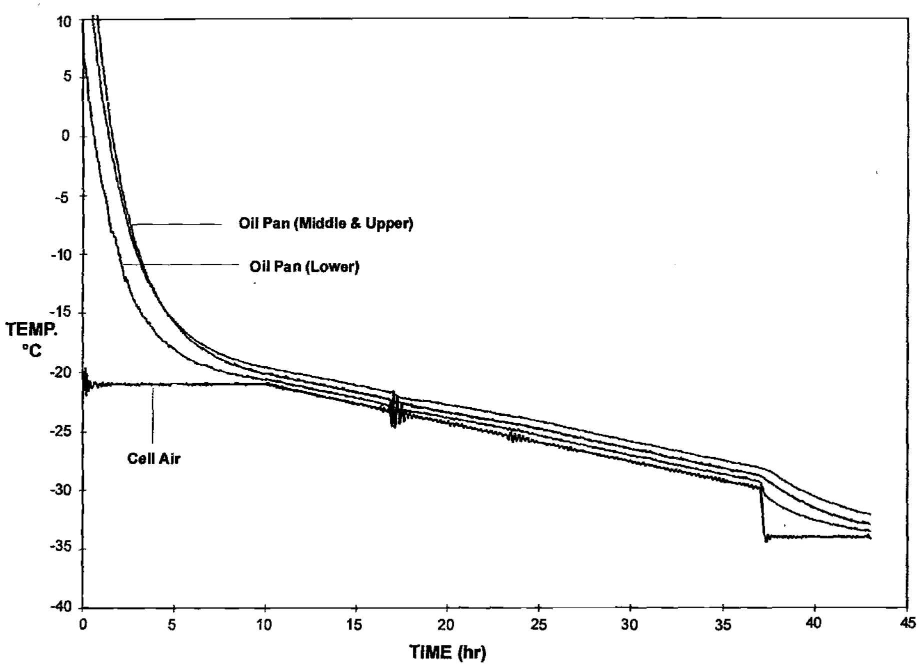


Figure 20 – *Alternate Sioux Falls Profile, 1.9L I4 Engine, LTEP 23*

Table 8 - *Phase II Pumpability Results on LTEP 23 in 1.9L I4, 2.3L I4 and 3.0L V6 Engines under Various Cooling Cycles*

			Pressurization Times, seconds	
Engine	Cooling Cycle	Oil Temp., °C	Pump Out, Time to 150 kPa	Near Galley, Time to 10 kPa,
1.9L I4	Rapid Overnight	-33.7	0.5	3.5
"	Mod. Sioux Falls	-33.0	0.5	4.0
"	Alt. Sioux Falls	-32.7	1.0	2.5
2.3L I4	Rapid Overnight	-33.8	0.5	5.5
"	Mod. Sioux Falls	-33.8	0.5	4.5
"	Alt. Sioux Falls	-33.0	0.5	4.5
3.0L V6	Rapid Overnight	-33.3	7.0	18.0
"	Mod. Sioux Falls	-32.7	7.5	18.0
"	Alt. Sioux Falls	-32.7	5.5	15.5

Table 9 - *Pumpability Results on LTEP 2 in 1.9L I4, 2.3L I4 and 3.0L V6 Engines under Rapid Overnight Cooling Cycle*

			Pressurization Times, seconds	
Engine	Cooling Cycle	Oil Temp., °C	Pump Out, Time to 150 kPa	Near Galley, Time to 10 kPa,
1.9L I4	Rapid Overnight	-33.9	1.0	7.0
2.3L I4	Rapid Overnight	-33.5	1.0	10.5
3.0L V6	Rapid Overnight	-34.0	7.5	16.0

Thus, the Phase II testing of an SAE 5W-30 oil with a gelation index of approximately 16 did not show any indications of air-binding pumpability problems under a variety of cooling profiles in these engines.

Conclusions

It is important to recognize that, with the exception of the 4.0L I-4 engine, all of the engines were of relatively modern design (1992-1993 vintage). Analysis of the near galley pressurization data had indicated that these modern engine designs had limiting pumping viscosities in the range of 71 to 131 Pa·s (as measured by D 3829) with an average of approximately 93 Pa·s. The older engine design (4.0L I-6), which showed higher minimum starting temperatures, had limiting pumpability viscosities in the range of 31 to 48 Pa·s with an average of approximately 43 Pa·s. This limiting viscosity is more consistent with earlier pumpability limits established in ASTM DS-57 [*5*].

The following conclusions can be made based upon limited testing in seven commercial gasoline engines of 6 Phase II multigrades. These experimental multigrades were selected because they demonstrated structure in certain bench tests such as TP-1 MRV and/or Scanning Brookfield procedures:

(1) Under appropriate cooling and operating conditions, air-binding pumpability could be generated in certain engine types.
(2) Air-binding conditions are strongly affected by oil height above the pick-up tube; very low oil charge exacerbates air-binding tendencies.
(3) Air-binding was observed only when significant structure was detected in the oils (>40 gel index, MRV Yield stress 70-105 Pa).

References

[1] Stambaugh, R. L., and O'Mara, J. H., "Low Temperature Flow Properties of Engine Oils," SAE Paper No. 821247.

[2] Smith, Jr., M. F., "Better Prediction of Engine Oil Pumpability Through a More Effective MRV Cooling Cycle," SAE Paper No. 831714.

[3] Henderson, K. O., Manning, R. E., May, C. J. and Rhodes, R. B., "New Mini-Rotary Viscometer Temperature Profiles That Predict Engine Oil Pumpability," SAE Paper No. 850443.

[4] *Cold Starting and Pumpability Studies in Modern Engines, ASTM Research Report RR-D02-1442*, American Society for Testing and Materials, West Conshohoken, PA, November 1998.

[5] *Low Temperature Pumpability Characteristics of Engine Oils in Full Scale Engines, ASTM DS-57*, American Society for Testing and Materials, Philadelphia, PA 19103, September 1975.

[6] Cold Starting and Pumpability Studies in Modern Engines - Results from the ASTM D02.07C Low Temperature Engine Performance Task Force Activities: Cold Start Testing, *Oil Flow Studies at Low Temperatures in Modern Engines,ASTM STP 1388*, American Society for Testing and Materials, West Conshohoken, PA.

[7] MacAlpine, G. A. and May, C. J., "Compositional Effects on the Low Temperature Pumpability of Engine Oils," SAE Paper No. 870404.

[8] Henderson, K. O. and Sicking, J. M., "The Effect of Shear Rate and Shear Stress on Viscosity Determinations at Low Temperatures for Engine Oils" SAE Paper No. 902091.

Christopher J. May,[1] Enrique F. De Paz,[2] Fred W. Girshick,[3] Kenneth O. Henderson,[4] Robert B. Rhodes,[5] Spyros Tseregounis,[6] and Lisa H. Ying[7]

Cold Starting and Pumpability Studies in Modern Engines - Results from the ASTM D02.07C Low Temperature Engine Performance Task Force Activities: Summary and Conclusions

---

Reference: May, C. J., De Paz, E. F., Girshick, F.W., Henderson, K. O., Rhodes, R. B., Tseregounis, S., and Ying, L. H., **"Cold Starting and Pumpability Studies in Modern Engines - Results from the ASTM D02.07C Low Temperature Engine Performance Task Force Activities: Summary and Conclusions,"** *Oil Flow Studies at Low Temperatures in Modern Engines, ASTM STP 1388*, H. Shaub, Ed., American Society for Testing and Materials, West Conshohocken, PA, 2000.

Abstract: Based upon the engine and oil types evaluated in the Low Temperature Engine Performance Task Force activities, it was found that (i) modern, fuel injected, gasoline engine designs, on average, start at lower temperatures than older types, and starting temperatures are independent of number of cylinders, configuration or displacement; (ii) CCS viscosities correlated with engine startability, but at considerably higher viscosities than in J300; (iii) an operating safety margin was observed between cold starting and pumpability; and (iv) these modern designs generally exhibited flow-limited behaviour and had limiting MRV pumpability viscosities in the range of 71 to 131 Pa·s with an average of approximately 93 Pa·s. On the basis of limited testing of 5 multigrade oils in the Phase II pumpability testing, it was found that air-binding pumpability could be generated in certain engine types, under appropriate cooling/operating conditions. Air-binding was only observed when significant structure was detected in the oils.

Keywords: cold starting, pumpability, light-duty engines, yield stress, gelation index, pumping viscosity, air-binding failure, flow-limited failure

---

[1] Senior Research Associate, Imperial Oil, Research Dept., P.O. Box 3022, Sarnia, Ont., Canada N7T 8C8.

[2] Project Leader, Consumers Union, 101 Truman Avenue, Yonkers, NY 10703-1057.

[3] HD Formulation Science Leader, Infineum USA LP, P.O. Box 536, Linden, NJ 07036.

[4] Vice President of Technology, Cannon Instrument Co., P.O. Box 16, State College, PA 16804-0016.

[5] Consultant, Infineum, USA LP, 11735 Normont Dr., Houston, TX 77070.

[6] Staff Research Engineer, General Motors Research Laboratories, P.O. Box 9055, Warren, MI 48090-9055.

[7] Senior Statistician, Infineum USA LP, P.O. Box 536, Linden, NJ 07036.

Keywords: cold starting, pumpability, lubricant rheology, viscosity, yield stress, gelation index, light duty engines

Introduction

This paper deals with the solicitation and selection of test lubricants for the cold starting and pumpability work carried out by the LTEP task force. Also, included is a discussion of the viscosity-temperature correlations developed for each lubricant; these were essential in order to allow the viscosities of the test oils to be estimated at the temperature of each cold start and pumpability engine test.

Test Oil Selection

The LTEP task force held several discussions during its formative stages to decide on the protocol to be used for the selection of test oils for the engine starting and pumpability studies. One approach was to concentrate only on oils which failed either the mini-rotary viscometer test at select temperature profiles or the Scanning Brookfield Viscometer test by gelation index. A drawback to this approach, however, was that it placed greater emphasis on the determination of engine cooling profiles that would fail potential problem oils, and less emphasis on one of the primary objectives of the task force which was to evaluate the starting and pumping characteristics of modern engines and determine safety margins between pumping and starting, rather than simply the rheology characteristics of oils. Another drawback was the anticipated difficulty in obtaining sufficient numbers and quantities of the potentially problem oils to evaluate.

An alternative approach, adopted as *Phase I*, was to request fully on-specification commercial oils. Such oils could be evaluated in starting and pumping studies at lower and lower temperatures until failures occurred. In this way, cost-effective engine cooling profiles could be used for testing, and pumping characteristics still could be related to the measurement of viscosity and yield stress in the mini-rotary viscometer, as was done in the 1975 ASTM program [*1*]. It was agreed further that a wide multigrade selection should be represented in the test program in order to meet primary task force objectives, and that the evaluation not be limited to a single type of additive or basestock. The most straightforward way of accomplishing this was by requesting commercially available oils, and by limiting the donation to one viscosity grade per company.. Subsequently a series of oils was donated by manufacturers and was assigned the following LTEP oil code numbers:

SAE Grade	0W-30	5W-30	10W-30	15W-40	20W-50	25W-30
LTEP #	1	2	3	5	6	7

LTEP 4 is missing from the above series. The amount of oil donated was insufficient for testing and it was dropped from the program. LTEP 8, a second SAE 15W-40, was received during the course of the test program; it was obtained primarily for heavy duty testing, which was never carried out. It should be noted that LTEP 7 is a straight grade 30 engine oil which also meets the low temperature requirements of a 25W; this oil was included to specifically address SAE's concerns regarding starting and pumpability of monogrades.

After these oils were obtained and the startability program had begun, the task force turned again to the question of testing potential problem oils. To do this, the task force encouraged the donation of oils which either did not meet the then-current SAE J300[8] pumping viscosity (TP-1 MRV) limits, or failed the Scanning Brookfield test by gelation index. Such oils were defined as "experimental oils", and in this report are identified as the *Phase II* test oils. Based on 100°C kinematic viscosities and CCS viscosities, these oils met the following J300 viscosity grades:

SAE Grade	15W-40	10W-30	5W-30	5W-30	10W-30	10W-30	10W-30
LTEP #	21	22	23	24	26	27	28

LTEP 21, an oil that failed an engine pumping test in the donor's laboratory, was not evaluated by the task force because it was received late in the program, was a heavy duty engine oil, and badly failed the MRV test. An offer to provide LTEP 25 subsequently was withdrawn by the prospective donor.

The last oils added to the Phase II series, LTEP 27 and 28 included two oils which failed the TP-1 MRV test by yield stress. During the course of the LTEP engine pumping studies, *flow-limited* pumping failures were observed, which are attributed to oils that have high low-shear-rate viscosities as measured in the Mini-Rotary Viscometer. However, there were no observations of *air-binding* failures of the type observed during some of the earlier ASTM 7-engine studies [*1*]. Air-binding failures are attributed to oils which show evidence of a yield stress in the Mini-Rotary Viscometer. It was determined that all of the Phase I oils either exhibited no yield stress, or a yield stress accompanied by a very high viscosity at the corresponding temperature. To determine if modern engines could still undergo air-binding failures, additional test oils were needed. A limited amount of LTEP 27, an oil which exhibited both failing yield stress and high viscosity under ASTM test method D 4684 conditions was added to the Phase II series late in the LTEP program. This oil had been reported to cause engine air-binding pumpability failure in laboratory engine tests [*2,3*]. LTEP 28 also was donated to the program at about this time. It is an oil which was designed to have significant yield stress and moderate viscosity in both the ASTM test methods D 3829 and D 4684 MRV at -25°C (see Appendix B in [*4*]). This combination of high yield stress and low viscosity was expected to produce only air-binding pumpability failure in the engines if cooled under the 2-day extended profile used in D 4684.

Initially it was decided that two reference laboratories would provide the bulk of the rheological data on the test oils needed to correlate with engine performance, to be supplemented by any data offered by the oil donors themselves. During the course of activities, however, it became apparent that additional rheological data were required, and these were provided by other LTEP member laboratories that volunteered their services - 13 labs in total [*4*]. Data obtained included ASTM test method D 445 kinematic viscosities, D 3829 MRV (overnight cooling), D 4684 MRV (TP-1 cycle), D 5133 Scanning Brookfield viscosity profiles and gel index, and D 5293 Cold Cranking

[8] SAE J300 MAR 92.

Simulator viscosities. MRV and CCS measurements at multiple temperatures were obtained for the test oils in order to allow effective correlation to the exact temperatures obtained in the engine test studies. All standard viscometric data on the test oils are summarized in Appendix B of the LTEP Research Report [*4*]. The data reported did not come about through a formal ASTM inter-laboratory program. Thus, if a lab chose to report a single average value, rather than independent values, that is what appeared in the summary results [*4*]. The ASTM LTEP Research report details results on all the rheological properties requested.

In addition to the standard rheology tests signified by an ASTM method, unique determinations also were made in Mini-Rotary Test apparati placed either in an engine test cell (exposed to the same cooling as the engine), or through software modification which controls the cooling profile in the instrument to simulate some of the alternate profiles used in the LTEP 20 series engine tests. These results are discussed within the framework of the Phase II low temperature pumpability paper as they were specific to particular engine test lab programs.

Test Oil Rheology

This section documents the fitting of rheological data (viscosity-temperature) to equations, thereby allowing interpolation to predict viscosity at the exact temperatures of the engine tests. In general, the viscosities were measured at integral temperatures, most often multiples of 5°C; the temperatures of the engine tests were usually recorded at multiples of 0.1°C.

For the raw viscosity-temperature data and detailed correlations, the reader is referred to Appendices B and F respectively in the LTEP Research Report [*4*].

Summary

Viscosity and yield stress were fitted to equations to allow interpolation to intermediate temperatures. The functional form of the equations are presented and explained, and the fitted coefficients are tabulated. A spreadsheet that automates the fitting procedure was also developed. For further information on the coefficients of the fitted equations, and some statistical diagnostics of the fits, the reader should consult the LTEP Research Report [*4*].

A separate fit was made for each of the 12 LTEP oils for each of seven viscometric tests, for a total of 84 equations. Viscosity data from D 3829 (Mini-Rotary Viscometer), D 4684 (Mini-Rotary Viscometer Temperature Profile 1), and D 5293 (Cold Cranking Simulator), were fit to a modified MacCoull-Walther-Wright equation that allows curvature. Yield stress data from D 3829 and D 4684 were fit to a logit function. Gelation Index and Gelation Index Temperature were arithmetically averaged for each oil, since they are not functions of temperature.

Description of Data

Viscosity-temperature data were collected from five test methods. Some methods give more than one result (*viz.*, Yield Stress and Viscosity from D 3829 and D 4684; Viscosity, Gelation Index, and Gelation Index Temperature from D 5133), and nine "types" of data were fitted. The test methods, their abbreviations as used in this section, and their descriptions are included in Table 1. For the purposes of this paper, only low temperature rheological properties will be reviewed.

Table 1 - *Types of Viscosity Data*

Abbreviation	Method	Description	Equation
CCS	D 5293	Cold Cranking Simulator	A
GI	D 5133	Gelation Index from SBT	D
GIT	D 5133	Gelation Index Temperature from SBT	D
KVC	D 445	Kinematic Viscosity	B
MRV	D 3829	Mini-Rotary Viscometer	A
SBT	D 5133	Scanning Brookfield Technique	A
TP-1	D 4684	Temperature Profile 1	A
YSM	D 3829	Yield Stress from MRV	C
YST	D 4684	Yield Stress from TP-1	C

where "Equation" denotes the functional form used to fit the data, as indicated in Table 2.

Table 2 - *Types of Viscosity-Temperature Equations*

Equation	Description
A	MacCoull-Walther-Wright with Quadratic Term (Centered)
B	MacCoull-Walther-Wright with Quadratic Term
C	Logit
D	Arithmetic Average (data not a function of temperature)

A more detailed, mathematical description of these equations follows later in this paper.

A goal was set to ensure that, for each oil in each test method, data were obtained for a minimum of four temperatures from at least three laboratories. Not every oil would be measured by every lab at every temperature (e.g., Round Robin-style), but sufficient overlap would be generated to check on the consistency of the measurements. In most cases, this minimum data requirement was exceeded [*4*].

Method for Fitting Viscosity Data

The fitting began with the MacCoull-Walther-Wright (MWW) equation, as published in Test Method D 341, "Viscosity-Temperature Charts for Liquid Petroleum Products." This test method and equation fit kinematic viscosity and temperature data to straight lines (of the form y = m*x + b) by use of a transformation. The form of the MWW equation, for kinematic viscosities above 2 mm^2/s, is:

$$Log[Log(\eta + 0.7)] = m * Log(T + 273.15) + b \qquad (1)$$

where

η is kinematic viscosity in mm^2/s,
T is temperature in degrees Celsius,
m is the fitted slope of the straight line,
b is the fitted intercept of the straight line, and
Log is the Naperian logarithm.

Low temperature viscosities are much greater than 0.7, and that constant was dropped for the low temperature data (retained for the kinematic viscosity data). Low temperature viscosities are expressed in the correct units for absolute viscosity, mPa·s.

The equation now becomes

$$Log[Log(\eta + a)] = m * Log(T + 273.15) + b \quad (2)$$

where

η is viscosity (in mPa·s for LT methods and mm^2/s for kinematic viscosity),
a is an empirical constant (= 0 for LT methods, 0.7 for kinematic viscosity),
m is the fitted slope (for each oil for each method), and
b is the fitted intercept (for each oil for each method).

For ease of nomenclature, the following abbreviations are introduced

$$W = Log[Log(\eta + a)] \quad (3)$$

$$X = Log(T + 273.15) \quad (4)$$

Equation (2) then becomes

$$W = m * X + b \quad (5)$$

which is the formula for a straight line in the transformed variables.

It was soon found that the data do not follow straight lines, even with the MWW transformation. A quadratic term was added to give the fit some curvature

$$W = q * X^2 + m * X + b \quad (6)$$

where

q is the fitted quadratic coefficient (for each oil for each method).

Numerically, these equations can be difficult to fit because of computer rounding errors, depending on the software used. This is due to the high correlation between "X" and "X^2" over the range spanned by the low temperature data. The data are first "centered" by subtracting a constant. This is mathematically equivalent to changing the origin of the Cartesian coordinate system.

$$W = q * (X - C)^2 + m * (X - C) + b \quad (7)$$

where C is a constant chosen to minimize the correlation between X and X^2, usually the arithmetic mean of the X data. Recognizing that C can be expressed as

$$C = Log(c + 273.15) \tag{8}$$

a physical meaning can be assigned to c. It is the (approximate) average temperature at which viscosity data were taken for each oil for each method.

To maintain simplicity, as much as possible, a rounded value of "c" was chosen for each test method, and applied to all oils (see Table 3).

Table 3 - *Centering Constants*

Method	Data Points	Minimum	Maximum	Median	Mean		Center
					Unweighted	Weighted	
CCS	137	-40	-5	-22.5	-20.9	-20.0	-20
SBT	224	-45.9	-5	-22.9	-21.8	-21.3	-20
MRV	179	-40.89	-5	-25.5	-28.2	-29.3	-30
TP-1	145	-45	-10	-27.5	-27.1	-27.3	-30

Recognizing that "X" and "C" are both logarithms, and that subtracting logarithms is the same as dividing, Equation 3.1.7 can alternatively be written

$$W = q * \left\{ Log\left[\frac{(T + 273.15)}{(c + 273.15)}\right]\right\}^2 + m * Log\left[\frac{(T + 273.15)}{(c + 273.15)}\right] + b \tag{9}$$

or as

$$W = q * X'^2 + m * X' + b \tag{10}$$

where X' is the centered and transformed temperature,

$$X' = Log\left[\frac{(T + 273.15)}{(c + 273.15)}\right] \tag{11}$$

The data were transformed using Equations (3) and (11), and plotted for inspection.

For each method, each oil was fitted to Equation (10) using the method of least squares. Plots of the raw data overlayed with the fitted lines were examined for "outliers" and points with high "leverage." Outliers are defined as points that don't seem to fit by visual observation; no statistical tests were used to identify outliers. Points with high leverage are points that affect the fitted parameters more than other points. These are commonly points at the highest or lowest temperature, and will be seen in subsequent graphs.

If a majority of data from other labs contradicted the point in question, it was dropped from the analysis. If more than one lab agreed about the questioned point, it was brought to the attention of the Task Force with a request for additional labs to re-measure the point, until a consensus could be reached.

The "goodness-of-fit" was determined both visually and statistically. The statistical coefficient of determination, R^2, was very high for all the fits. Only two cases were less than 0.9, and this was considered an inadequate parameter to judge the fit. The root mean square residual error (RMSRE) was calculated for each fit. This parameter, for a regression analysis, is analogous to the standard deviation for simple arithmetic averaging. The RMSRE was compared to the standard deviation for reproducibility, σ_R, for each test method. If the RMSRE is less than or approximately equal to σ_R, that means the multiple-temperature data are as consistent as the test method is capable of measuring. In all cases, the form of the viscosity-temperature fit, Equation (9), is different from the transformation used for the analysis of reproducibility in the test methods. The reproducibilities from the test methods were translated to the functional form of Equation (9), which leads to the σ_R's being represented by a range rather than a constant number [4].

Adjustment for Yield Stress - For the two test methods that measure yield stress in addition to viscosity, D 3829 (MRV) and D 4684 (TP-1), an adjustment was made to the measured viscosity before fitting. The physical situation is illustrated in Figure 1.

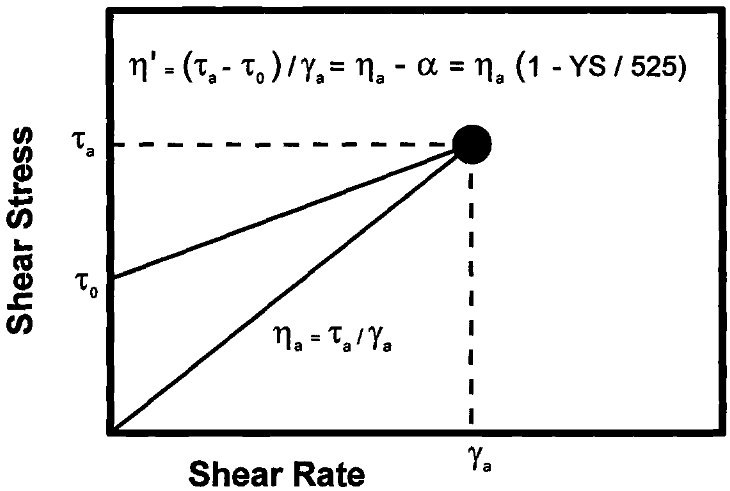


Figure 1 - *Viscosity Adjustment for Yield Stress*

The test methods measure an apparent viscosity, which is the ratio of apparent shear stress to apparent shear rate. The shear stress is applied by a weight, and the shear rate is calculated from the observed rotational speed.

$$\eta_a = \frac{\tau_a}{\gamma_a} \tag{12}$$

where η_a is the apparent viscosity, τ_a is the apparent shear stress, and γ_a is the apparent shear rate.

When a yield stress is present, the actual stress available to shear the oil is reduced. As seen in Figure 1, the measured data can be modified

$$\eta = \frac{\tau_a - \tau_0}{\gamma_a} = \frac{\tau_a}{\gamma_a} - \frac{\tau_0}{\gamma_a} = \eta_a - \left(\frac{\tau_a}{\gamma_a}\right)\left(\frac{\tau_0}{\tau_a}\right) = \eta_a - \eta_a\left(\frac{\tau_0}{\tau_a}\right) = \eta_a\left[1 - \frac{YS}{525}\right] \quad (13)$$

where η is the true (adjusted) viscosity, τ_0 is the yield stress, and YS is the measured yield stress in Pascals. (Note: The test methods normally apply a shear stress of 525 Pa for viscosity determinations.)

Method For Fitting Yield Stress

Examining graphs of yield stress vs. temperature suggest a logit equation would be an appropriate functional form for fitting:

$$YS = \frac{A}{(1 + e^{[(T-B)/C]})} \quad (14)$$

where

YS = yield stress, Pa,
T = temperature, °C,
A, B, C = fitted parameters.

The fitted parameters can be assigned physical meanings. The parameter "A" represents the asymptotic (highest) yield stress at the lowest temperatures. Since the highest yield stress that can be measured by D 3829 or D 4684 is 525 Pa, the fitted values of "A" were constrained to be no greater than 525 Pa. There were seven (out of 24) cases where "A" was fitted to 525 Pa. The presence of the constraint makes very little difference to the visual appearance of the fitted curve, but makes more sense physically.

The parameter "B" is related to the temperature below which yield stress begins to rise. The parameter "C" provides a "scale," representing how fast yield stress increases with decreasing temperature.

The form of Equation 12 forces the fit of yield stress to increase monotonically with decreasing temperature. It has been reported [5] that it is possible, as temperature decreases, for yield stress to increase then decrease. This behavior was not observed experimentally for any of the LTEP oils.

Graphs and Discussion

Representative graphs of the transformed raw data and the fitted equations are shown in Figures 2 - 7, and discussed below. All the low temperature viscosity graphs are plotted to the same scales to facilitate comparisons. The x-axis range of 2.36 - 2.44 in Log(Temperature, °K) corresponds to roughly -44°C to +2°C. The y-axis range of 0.4 to 0.8 for Log[Log(Viscosity)] corresponds to roughly 325 to 2 million mPa·s.

Cold Cranking Simulator (CCS), ASTM D 5293 - Typical plots of CCS viscosities and fitted equations are contained in Figure 2. All of the CCS viscosities showed almost straight-line behaviour, with very slight curvature. All the R^2's are above 0.998, and the RMSRE's are within the range of test method reproducibility.

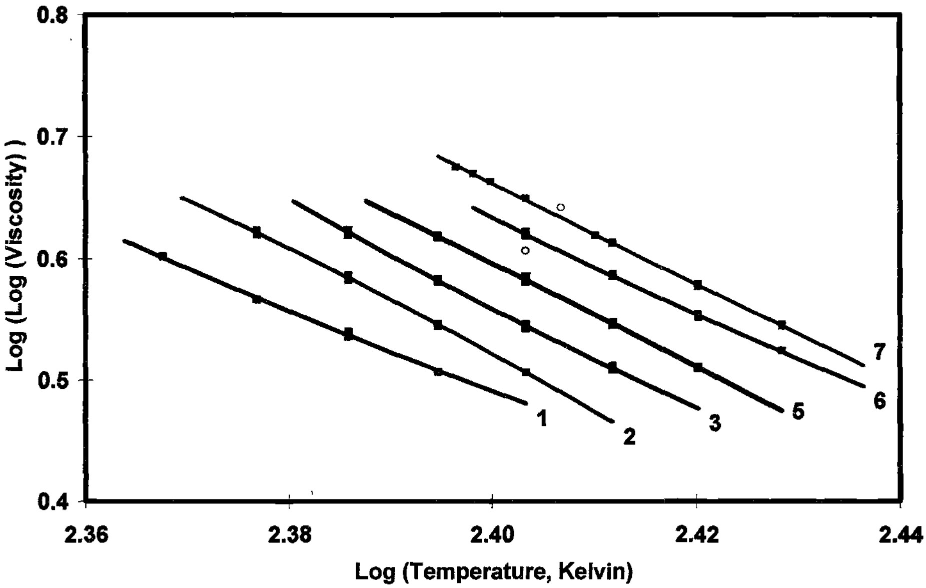


Figure 2 - *LTEP CCS Viscosity-Temperature Data for LTEP 1-7*

Mini-Rotary Viscometer (MRV), ASTM D 3829 - Examples of the MRV viscosities and fitted equations are shown in Figure 3 for LTEP 1-7. Viscosities were adjusted for yield stress *per* Equation 13. Outlier points omitted from the fit, but plotted for information are highlighted by arrows; these included LTEP 2, 3, 5 and 23 (latter not shown [*4*]). All of the MRV viscosities showed some slight curvature, with LTEP 26 the most pronounced; that oil had a point of very high "influence," i.e., one point almost completely determines the curvature of the line [*4*].

All the R^2's were above 0.976, and the RMSRE's were within a factor of two of test method reproducibility. Mean values for each oil at each temperature are shown in Table 4.

Scanning Brookfield Technique (SBT), ASTM D 5133 - Examples of the SBT viscosities and fitted equations are shown in Figures 4 and 5. While there was good agreement between labs for some oils, unresolved discrepancies were noted for others. For example, LTEP 6 (Fig. 4) had three labs giving relatively straight-line behaviour, and

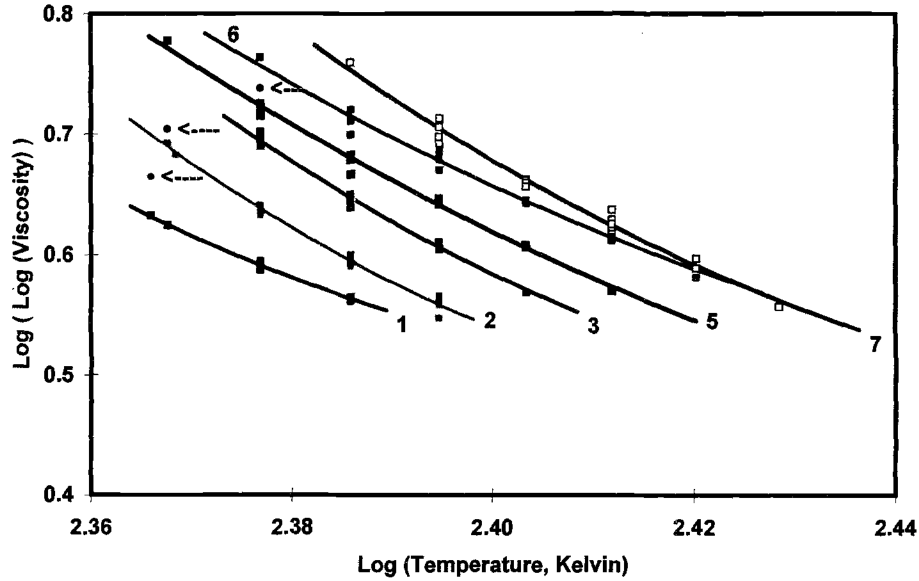


Figure 3 - *LTEP D 3829 MRV Viscosity-Temperature Data for LTEP 1-7*

Table 4 - *MRV (D 3829) Mean Values*

	MRV (D 3829) Average Viscosity at Temperature, °C (mPa·s)							
Oil	-40	-35	-30	-25	-20	-15	-10	-5
LTEP01	15,908	7,858	4,508					
LTEP02	82,294	22,040	8,559	4,106				
LTEP03	113,767	88,762	26,444	11,142	5,000			
LTEP05	973,177	187,532	58,873	24,925	11,126	5,210		
LTEP06		427,101	133,906	59,058	24,975	13,596	6,360	
LTEP07			552,263	111,263	36,970	17,157	8,158	4,000
LTEP22	162,292	47,207	18,201	7,956				
LTEP23	135,304	32,457	10,772	4,063				
LTEP24	91,008	30,224	11,891	5,501				
LTEP26		51,127	21,597	11,148	5,889			
LTEP27	344,106	105,086	37,482	14,984	7,381			
LTEP28	126,620	42,970	16,572	7,697	4,000			

Note: Mean values calculated by averaging transformed data, log[log(viscosity)], after correcting for yield stress, per Equation 13.

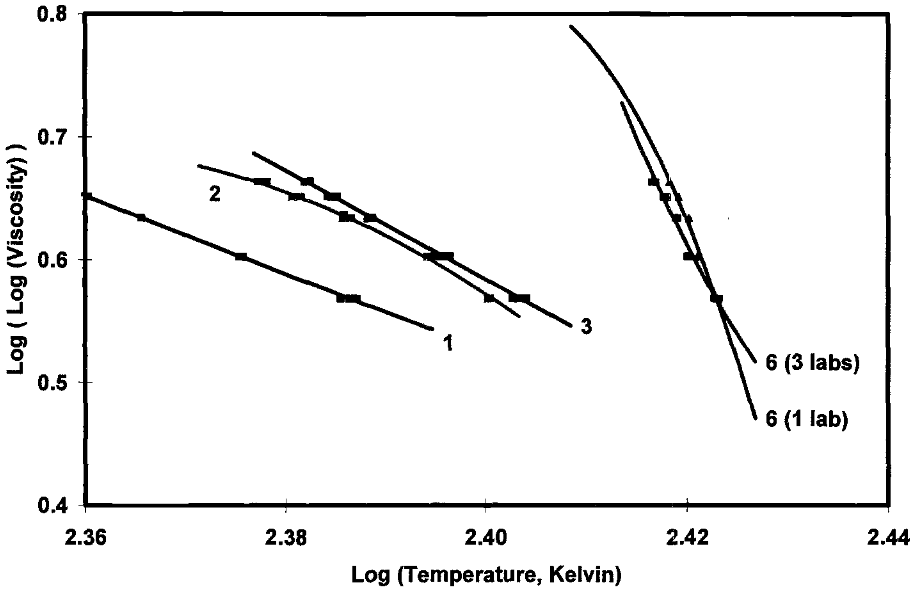


Figure 4 - *LTEP D 5133 SBT Viscosity-Temperature Data for LTEP 1-3, 6*

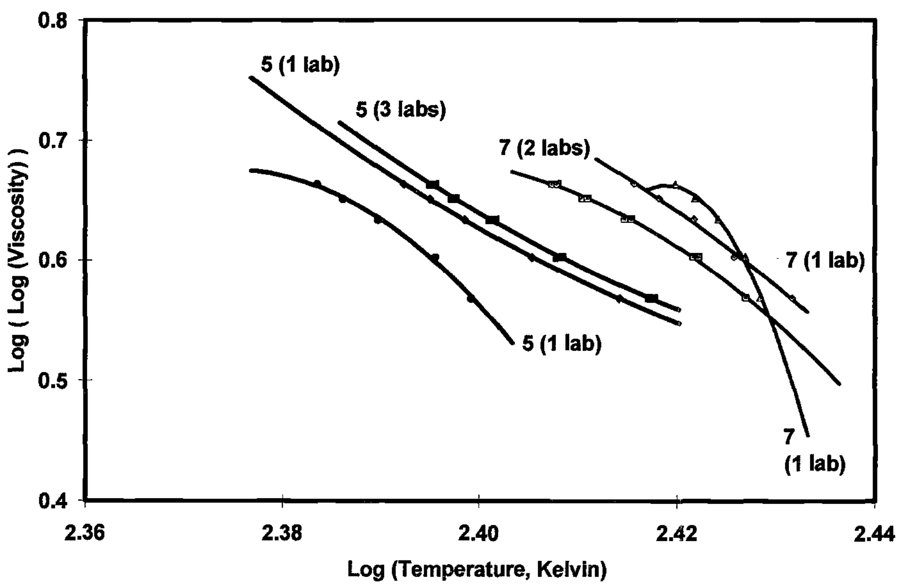


Figure 5 - *LTEP D 5133 SBT Viscosity-Temperature Data for LTEP 5, 7*

one lab showing curvature. Differences between labs were even greater for LTEP 5 and LTEP 7 (Fig. 5). There was no correlation among the labs; i.e., the same lab was not always severe for these three oils.

Many of the oils showed "asymptotic" behaviour at the lower end of their temperature range. This was assumed to be an artifact of reaching the upper limit of instrumental response.

All the R^2's were above 0.87, except LTEP 24, which was 0.78. The SBT test method reports the temperature for a given viscosity, and the equations fit viscosity as a function of temperature. This makes the standard regression diagnostics difficult to compare to the published test precision.

Temperature Profile 1 (TP-1), ASTM D 4684 - Examples of the TP-1 viscosities and fitted equations are in Figures 6 and 7. Viscosities were adjusted for yield stress *per* Equation (13). LTEP 7, 23, and 26 had outlier points that were omitted from the fit, but plotted for information [*4*] (e.g., see Fig. 6). LTEP 28 had a point of very high "influence," i.e., this one point completely determined the curvature of the line. Without that one point, the curve would be linear below -30°C, as shown (Figure 7).

LTEP 6 had discrepancies among the labs, and in order to decide on the final fit, the behaviour of the other oils was considered [*4*]. All of the TP-1 viscosities showed some slight curvature, with LTEP 5 and 27 the most pronounced. Both concave upward and concave downward curvature was seen. All the R^2's were above 0.95, and the RMSRE's were within a factor of two of test method reproducibility, except LTEP 2, 5, 6, 24, and 28. Mean values for each oil at each temperature are shown in Table 5.

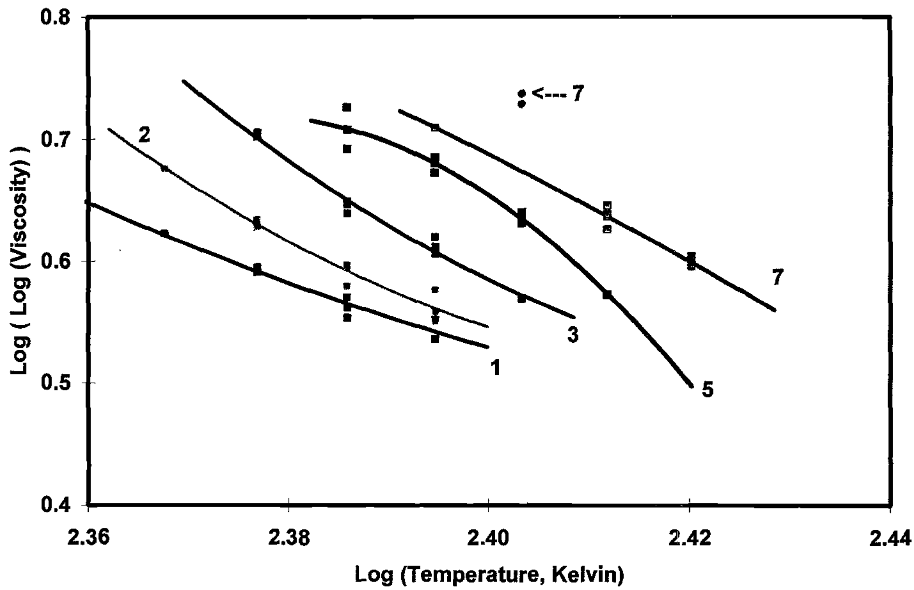


Figure 6 - *LTEP TP-1 MRV Viscosity-Temperature Data For LTEP 1-3, 5 & 7*

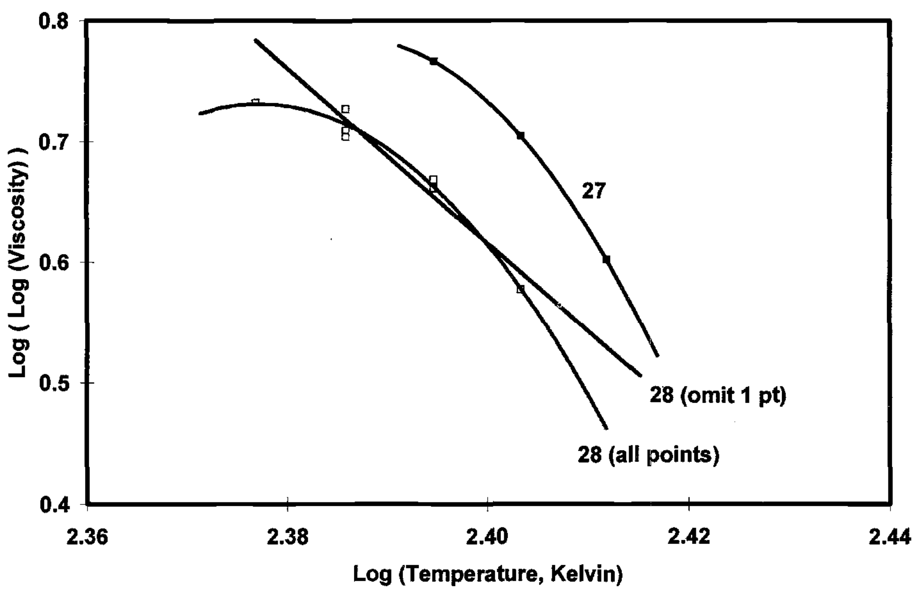


Figure 7 - *LTEP TP-1 MRV Viscosity-Temperature Data For LTEP 27 & 28*

Table 5 - *MRV TP-1 (D 4684) Mean Values*

	MRV TP-1 (D 4684) Average Viscosity at Temperature, °C (mPa·s)							
Oil	-45	-40	-35	-30	-25	-20	-15	-10
LTEP01	31,000	15,400	8,173	4,399	3,125			
LTEP02		53,700	18,752	7,825	4,354			
LTEP03			111,879	26,375	11,929	5,050		
LTEP05				129,696	59,788	20,769	5,454	
LTEP06					105,401	68,800	16,521	17,900
LTEP07					130,902		21,381	9,300
LTEP22			59,431	22,103	9,937	4,865		
LTEP23		91,440	40,893	11,056	4,585			
LTEP24		131,937	36,994	13,610	7,423			
LTEP26			96,343	24,464	13,843	6,300		
LTEP27					686,900	115,933	10,000	
LTEP28			248,613	148,273	40,651	5,966		

Mean values calculated by averaging transformed data, log[log(viscosity)], after correcting for yield stress, per Equation 13.

Yield Stress by MRV and TP-1 (YSM & YST), ASTM D 3829 & D 4684 - An example of yield stress and fitted equations are shown for LTEP 6 in Figure 8.

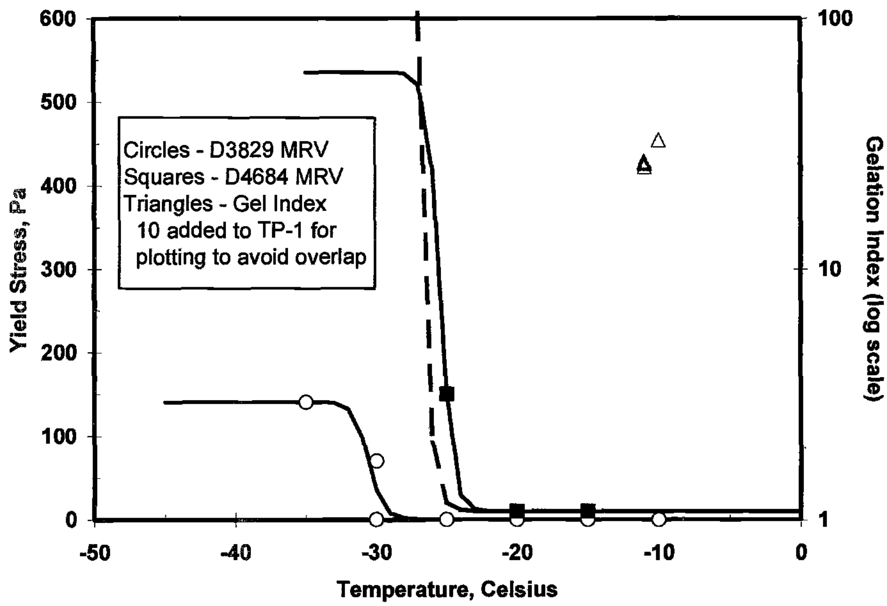


Figure 8 - *Yield Stress and Gel Index Plots for LTEP 6*

The "quantized" nature of yield stress measurements ensures relatively poor fits to any equation. The two test methods were designed to detect the presence of yield stress, rather than quantify the exact amount. In that sense, these data are being "misused." The logit function does an adequate job, but discrepancies among labs were evident. For example, for the TP-1 on LTEP 24, one lab did not detect any yield stress, while the other labs did (Figure 9). That is reflected in the fitted curve being somewhat below where one might draw it "by eye." The worst example was TP-1 data for LTEP 27, where almost half the labs detected no yield stress, and half the labs detected the maximum yield stress (525 Pa).

Figure 10 shows the average yield stress by the two methods, for each oil at each temperature. All but one point (LTEP 3 at -35°C) are above the one-to-one line, indicating TP-1 is the more severe method.

Gelation Index and Gelation Index Temperature (GI & GIT), ASTM D 5133 - Gelation Index and Gelation Index Temperature were superimposed on the Yield Stress graphs as illustrated in Figures 11 and 12 [4]. The pooled reproducibility for Gelation Index is 38%, which agrees favorably with the test method precision (43%). The pooled reproducibility for Gelation Index Temperature is 4.4°C, for the oils whose average Gel-

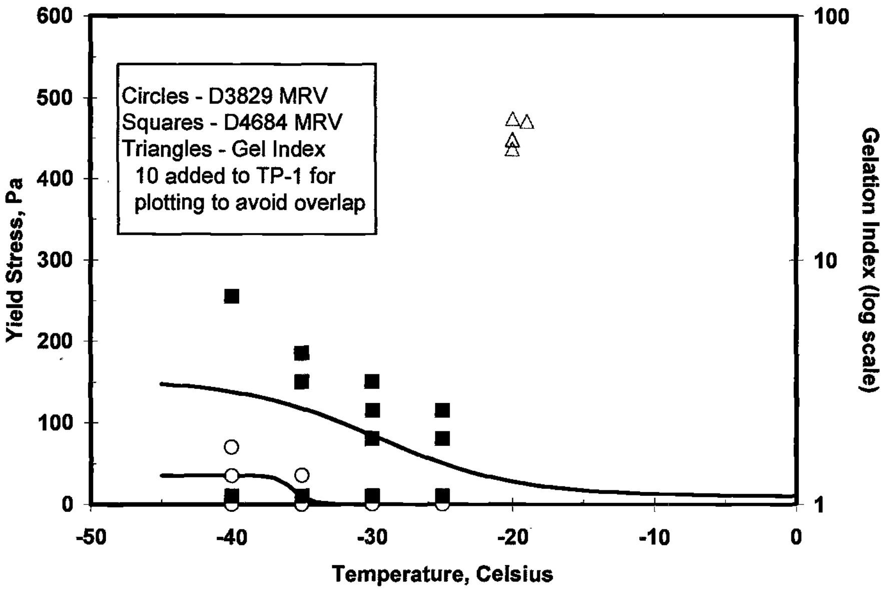


Figure 9 - *Yield Stress and Gel Index Plots for LTEP 24*

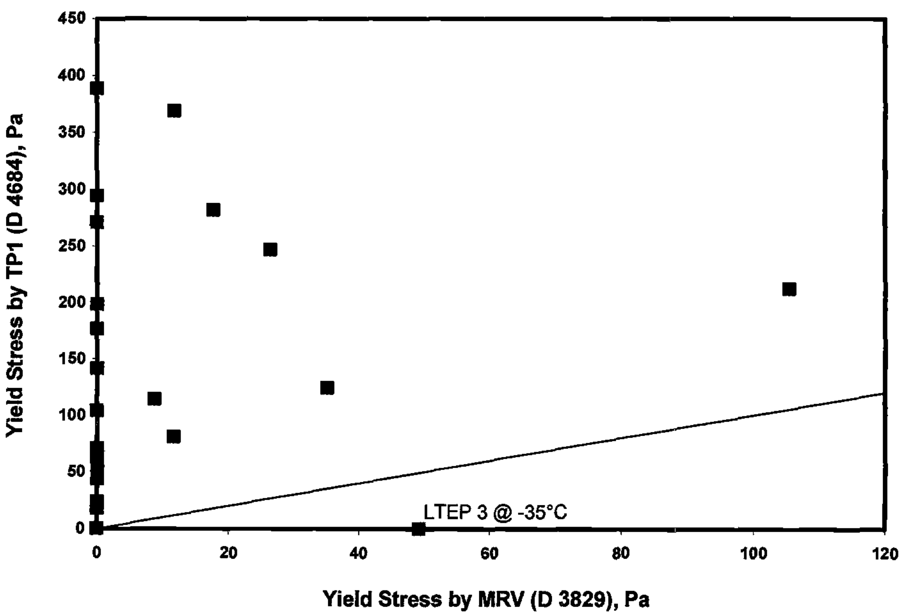


Figure 10 - *LTEP Comparison of Yield Stress by Two MRV Methods*

ation Index is above 6 (excluding LTEP 1 and LTEP 3), which agrees favorably with the test method precision (5°C).

While Yield Stress is a function of temperature, Gelation Index is a single value for an oil and occurs at the Gelation Index Temperature. The Yield Stress at that temperature can be calculated using the equations and fits described earlier in this paper. Figure 11 is a cross-plot of Gelation Index and the fitted Yield Stress at the Gelation Index Temperature. The dashed line shows the current pass/fail regions for yield stress (SAE J300 maximum less than 35 Pa) and Gelation Index (API GF-2 maximum 12). (Note: symbol types and sizes have been adjusted to show where points from the two different MRV methods overlap.)

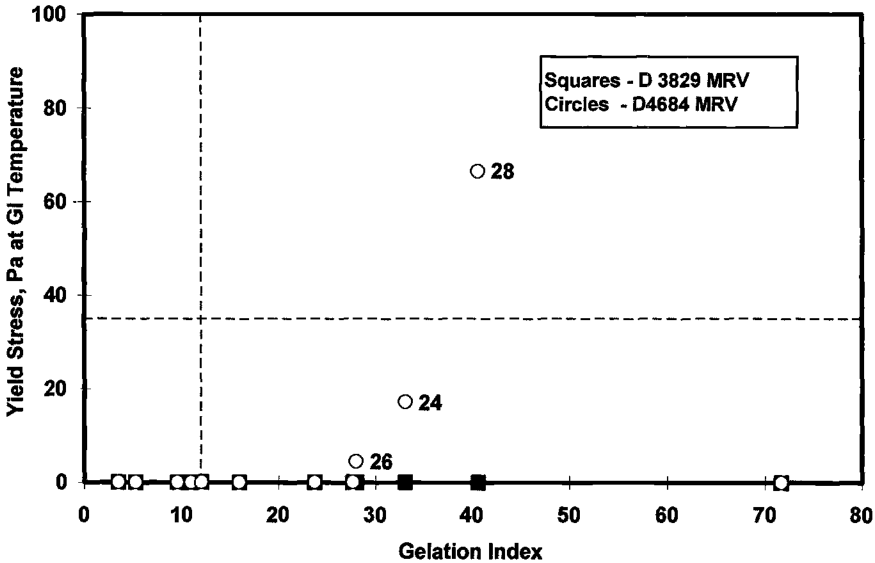


Figure 11 - *Gelation Index and Yield Stress at Gel Index Temperature*

At the Gelation Index Temperature, only oils LTEP 24, 26, and 28 showed detectable yield stress. Four oils showed no detectable yield stress even with Gelation Indices between 16 and 75. Only LTEP 28 would fail both TP-1 and Gelation Index.

Figure 12 is a similar plot, but showing yield stress at the SAE Grade temperature for yield stress (e.g., -30°C for an SAE 10W-xx). Four oils would pass both TP-1 yield stress and Gelation Index. Five oils would fail both TP-1 yield stress and Gelation Index. Three oils (LTEP 6, 22, and 23) would pass TP-1 yield stress but fail Gelation Index.

Session II

Theodore W. Selby[1]

Pumpability - Past Accomplishments; Present and Future Challenges

---

Reference: Selby, T. W., "**Pumpability - Past Accomplishments; Present and Future Challenges,**" *Oil Flow Studies at Low Temperatures in Modern Engines, ASTM STP 1388,* H. Shaub, Ed., American Society for Testing and Materials, West Conshohocken, PA, 2000.

Abstract: The possible problem of inadequate low-temperature engine oil pumpability was considered in the early '60s and grew into prominence in the late '60s and early '70s with the success of the automotive industry in improving low-temperature engine startability. Reported incidents of field pumpability problems in the early '70s led to an extensive ASTM study using several cold-rooms and a number of reference oils. This study confirmed the previously anticipated existence of two forms of pumpability problems -- flow-limited pumpability caused by higher viscosities and air-binding pumpability caused by engine oil gelation under certain cooling conditions.

Of these two forms of engine pumpability failure, air-binding was the primary threat to engine life. This was a consequence of the fact that some engine oil formulations could be induced to gelate at temperatures well above viscosity-limited temperatures. Despite these extensive cold-room studies and the development of a Mini-Rotary Viscometer bench test closely simulating the cold-room results, the work did not prepare those studying the problem area for the disastrous winter of 1980-81 when several million dollars of engines failed in the field as a result of air-binding oil effects.

In renewed efforts to develop viable bench tests correlating to these field-failing engine oils representing real world conditions, two instruments emerged: one in 1982 called the Scanning Brookfield Technique and the second, a further adaptation of the original MiniRotary Viscometer (called the MRV TP-1), in 1985.

Another recent ASTM cold-room study has confirmed improved startability and brought into question whether or not modern engines are less prone to air-binding.

At the same time, new pressures on both automotive and heavy-duty diesel engines have just come to attention as a consequence of long drain intervals and their effects on engine oil oxidation. It has been shown that such intervals can have serious effects on the engine through the coupling of both viscosity-limited and air-binding characteristics. In a similar manner, high soot loading in diesel engines recently redesigned to reduce oxides of nitrogen has produced further low-temperature pumpability problems and the associated needs to understand the chemistry and rheology of such oils at these temperatures and to find ways of reliably and repeatably measuring these phenomena.

Key Words: Pumpability, oxidation, air-binding, viscosity-limited, flow-limited, Brookfield Viscometer, Gelation Index, Gelation Index Temperature, D 5133, Scanning Brookfield Technique, MiniRotary Viscometer, cold-room engine tests

---

[1] Director R&D, Savant, Inc., 4800 James Savage Road, Midland, MI, 48642.

Early Studies

General Considerations

The ability to pump the engine oil through the automotive engine is an unquestioned need by virtually anyone vaguely familiar with the operation of that mechanism. Adequate pumpability is assumed by the operator of the automobile, bus or truck and it is an expensive shock to the owner when this does not occur. Absence of adequate pumpability has serious implications not only for the engine but for the owner and, if warranty provisions are present, for the engine manufacturer and/or lubricant supplier.

Over a period of three decades following World War II, successful efforts by both automotive and petroleum engineers made the passenger car more dependable in winter climates [*1 (summary reference work)*]. Startability was the focus of that work. However, as this impediment to automotive use at low temperatures was removed, the question of engine oil pumpability arose.

A 1963 paper on low-temperature engine startability and engine oil pumpability anticipated potential problems [*2*]. Among other aspects discussed, the paper pointed out the vulnerability of the engine to inadequate pumpability. The paper underscored the importance of the latter and speculated that pumpability failure could occur by either of two mechanisms: viscosity-limited flow or oil gelation. Of the two, the latter was considered most likely to lead to air-binding of the oil pump. These mechanisms were later demonstrated by cold-room studies and bench tests, as will be discussed.

However, other forms of pumpability problems have very recently arisen with the greater overuse of automotive lubricants in modern, smaller, higher-powered engines as well as the effect of soot in very highly soot-laden, heavy-duty engine oils. These kinds of pumpability problems may occur even at relatively mild ambient temperatures.

Years ago, loss of oil pumpability at low and normal starting temperatures were generally associated with some form of engine mechanical malfunction such as a stripped distributor gear. Today, with the mechanical reliability of the oil pumping systems of modern passenger car and heavy-duty engines, engine oil gelation, oxidation, sooting and their combinations are the major causes of poor oil pumpability and engine damage. As such, the three phenomena are major challenges to additive development and the proper formulation and blending of engine oils. The rheology of all three effects will be discussed in this paper.

First Major Study of Pumpability

In view of the relative lack of knowledge of engine oil pumpability effects at low temperatures in the early '70s, a major study in cold-room engine tests by cooperative work of automobile manufacturers, engine oil producers, and additive manufacturers was conducted under the aegis of the ASTM. This study, reported in 1975 [*3*], showed that pumpability could become a problem in the two ways predicted about a decade earlier [*2*]. Not surprisingly, it was confirmed that the most apparent way to lose engine oil pumpability was for the viscosity to reach a value at which the oil would not flow to the pump fast enough to satisfy the engine requirements for lubrication. For a given engine, once the limiting pumpability temperature was known, this viscosity-limited (or "flow-limited") response could be readily predicted by measuring an oil's viscosity.

The second form of limited pumpability, called "air-binding" was also confirmed in the ASTM study and was, as speculated earlier, unpredictable. Air-binding seemed to be a function of the tendency of some oils, under certain cooling conditions, to form an association of molecules -- a "structure" or "network" -- in which the associated molecules would collectively exert resistance to relatively low shear stresses but flow more readily at higher stresses. This is a form of flow behavior often associated with gelatinous matter and, for this reason, was called "gelation" in describing the condition. Interestingly, with certain cooling conditions, gelation could occur at temperatures at which -- under other cooling conditions -- the oil would flow with no evidence of gelation. Prediction of this form of pumpability problem could be made only by appropriate bench tests.

Development of the MRV ASTM D 3829

Following the ASTM cold-room pumpability study, cooperative work by passenger car engine manufacturers, petroleum companies, additive manufacturers and an instrument company, made possible a bench test called the MiniRotary Viscometer (MRV).

The MRV gave generally high correlation with these engine tests as shown in Figure 1 in which the 7-Engine Average Borderline Pumping Temperature (BPT) was compared with the MRV BPT [*3,4*]. The Coefficient of Determination, R^2, was 0.983 with 1.0 being perfect correlation; the slope was 1.05 with 1.0 again being perfect; and the intercept was 0.74°C with 0.00°C being perfect. Three oils showed yield stress: PRO-05, PRO-09, and PRO-10.

With such promise as an effective screening tool, in 1979, after a successful round robin in the ASTM, the MiniRotary Viscometer was made ASTM Standard Test Method for Predicting the Borderline Pumping Temperature of Engine Oil (D 3829).

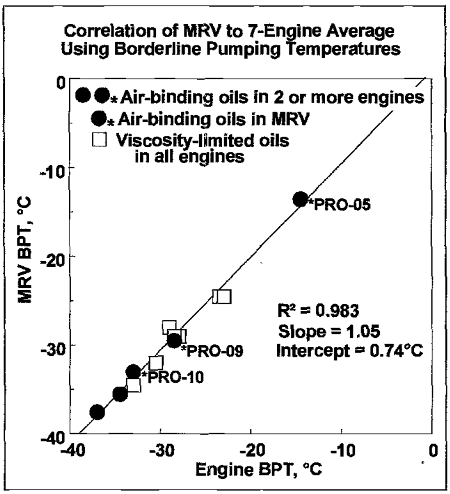


Figure 1

Pumpability Problems - Sioux Falls, South Dakota

Confidence in the knowledge gained in the '70s ASTM cold-room results and the anticipated ability to predict failure in the field with the ASTM D 3829 bench test was seriously eroded during the winters of 1980-81 and 1981-82[*1*]. Massive field failures occurred during the 1980-81 winter in Sioux Falls, South Dakota caused by air-binding oils a number of which were unrevealed using the then just-published ASTM D 3829 method. As a consequence of this epidemic of failures for no obvious reason at the time, technical efforts to understand the causes of pumpability failure of the oils became intense as soon as these field oils became available for study in 1981.

Fortunately for the whole effort to define pumpability and to correct the causes of the field failures, a seminal paper by Stambaugh and O'Mara in 1982 demonstrated in the cold room the cause of the field failures in Sioux Falls [*5*]. The paper showed the impact of very subtle changes in cooling conditions on the formation of gelation in engine oils and the catastrophic engine effects of these changes.

Their work also revealed a two-step temperature process in the formation of gelation that would generate such impact. The first step was the formation of gelation at a constant moderately low temperature over a period of seven to ten hours followed by a more rapid decrease in temperature of at least 5°C. They attributed the phenomenon to "both a critical wax nucleation and crystal phase growth" that were necessary to develop such pumping failure. This cooling pattern became known as the "Sioux Falls Cycle".

Yet, in a July 30, 1985 letter by Stambaugh to the author regarding a commercial oil detected by the Scanning Brookfield Technique (to be discussed in the following section), he showed that some oils passing the Sioux Falls Cycle if tested in a different cooling protocol would produce catastrophic engine failure.

New Instrumental Approaches

MiniRotary Viscometer - Temperature Profile 1 (TP-1)

Modification studies - Dedicated work by several investigators sought modifications of the MRV to reestablish the applicability of that bench test [*6, 7*]. Measuring the oil gelation with greater sensitivity and precision was considered of critical importance in view of the previous failure to identify field-failing oils.

By 1985, a method called TP-1 was suggested [*8*] which correctly identified the field-failing oils as air-binding. This method required a much slower cooling rate in order to gain yield-stress response from all field-failing oils including PRO-26 and PRO-29 which in earlier breakthrough work with the MRV by Smith had proven refractory (7).

Correlation with ASTM engine tests - When the TP-1 method was applied to the '70s PRO oils, the results shown in Figure 2 were obtained. Comparing these results to those previously shown in Figure 1 for the MRV, the TP-1 protocol worked well with oils identified by the engines as viscosity-limited but not as well on air-binding oils.

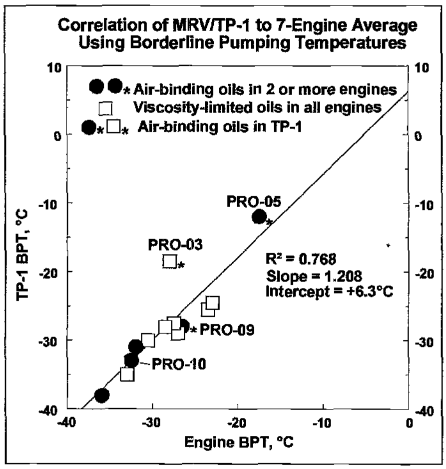


Figure 2

ASTM acceptance - The ability to identify the field-failing engine oils, however, encouraged an ASTM round robin on the MRV TP-1 protocol. Results showed that the method gave reasonable precision regarding viscosity-limited oils but because of relatively poor precision particularly at low yield stress values, classification of air-binding oils was limited to a simple go/no-go test at a shear stress of 35 Pascals, the minimum shear stress used in developing the method [*8*]. The method was accepted in 1987 as ASTM Standard Test Method for Determination of Yield Stress and Apparent Viscosity of Engine Oils at Low Temperature (D 4684).

Scanning Brookfield Technique

First work - The first instrumental procedure to indicate that the Sioux Falls field failures were primarily oil-related rather than engine-related was the Scanning Brookfield Technique (SBT) [*9*]. The technique was a constant speed, rotational viscometry method developed in 1981 by the author and his associates and later described in an oral discussion [*10*] of the aforementioned paper by Stambaugh and O'Mara [*5*].

The Ogee Curve - The SBT protocol is unusual in that it slowly cools the oil sample at 1°C/hr while the viscosity is continuously measured at slow rotor speed -- a temperature-scanning viscometric approach. As sketched in Figure 3, during first SBT tests of a field-failing oil, it was found that the normal exponential viscosity-temperature curve of a well-behaved oil, **a**, was considerably different from that of a gelated oil, **b**, whose curve was distorted into an ogee shape. For a field-failing oil, the temperature at which this distortion began corresponded to the "soak temperature" reported independently by Stambaugh and O'Mara on a very similar oil [*5*]. They speculated that exposure to this temperature gave a "wax nucleation" phase followed by a "crystal growth" phase after a 5°C drop in temperature.

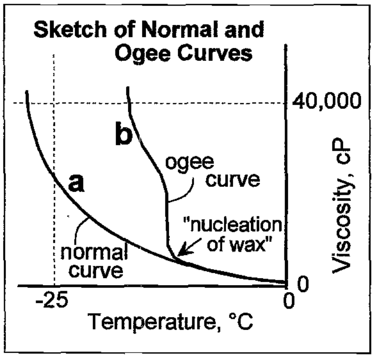


Figure 3

SBT correlation with field-failing oils - With success in detecting marked difference between non-gelating and gelation-forming engine oils, the technique was applied to all the field-failing oils available. Results on field-failing oils in Figure 4 are plotted in comparison to the form of a normal, non-gelating oil. All of the field-failing oils showed the ogee form of curve. Moreover, the method showed differences among the oils in the degree to which they developed the ogee form. Field-failing oils PRO-26 and PRO-29a (the latter of which was known from cold-room tests to be only a borderline failure) were shown to have less pronounced ogee curves. (These same two oils were found challenging in the development of the MRV TP-1 method [*7,8*] because of the need to develop a protocol under which they would both fail.)

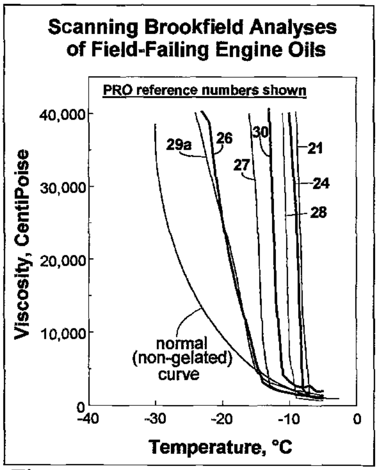


Figure 4

As a test of the ability of the SBT to detect the field-failing oils, when an SAE 15W-40 engine oil with a sharp ogee curve was found on the market, it was subjected to cold-room engine pumpability tests by Stambaugh and his associates. In these tests, the oil did not fail using the Sioux Falls cycle but did fail catastrophically using the TP-1 cooling protocol.

Thus, it became evident that 1) bench tests could predict air-binding pumpability failure even when 2) previously successful cold-room protocols might fail to respond. The oil became PRO-30 and its ogee curve -- initiating at about -10°C -- is also shown in Figure 4.

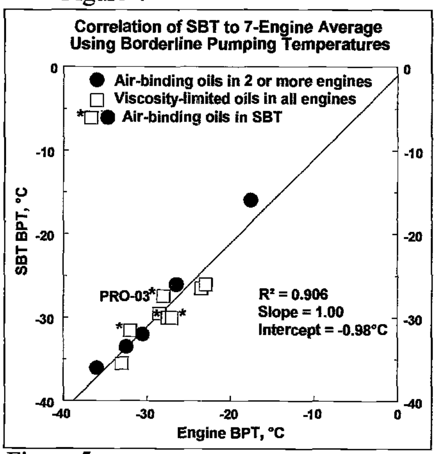


Figure 5

Engine Cold-Room Test Correlation - Figure 5 from a summary paper on the method [*9*] shows the correlation with the ASTM seven-engine study. The values for air-binding oils applied the 5°C correction required to obtain air-binding pumpability failure in the engine tests of Stambaugh and O'Mara. This temperature correction was related to a "crystal growth phase" development [*5*]. Several of the early PRO oils in this SBT study showed ogee curves on oils that were, however, indicated by the engines to be flow-limited. The explanation for this difference in response was that the ASTM cold-room tests dropped the engine temperature fairly rapidly and may have prevented the development of the gelation that slower, field-failure-type cooling would have revealed. One of these oils was PRO-03, also noted in Figure 2 to have a yield stress in the MRV TP-1.

Figure 5 shows that correlation between the 7-engine average and the SBT in regard to Borderline Pumping Temperature was comparatively good with an R^2 value of 0.91 and all values falling about a best line with slope of 1.00 and intercept close to zero (0.98°C). All oils indicated to be air-binding in the cold-room showed SBT ogee curves.

ASTM Acceptance - In 1990, the SBT was accepted as ASTM Standard Test Method for the Low Temperature, Low Shear Rate, Viscosity/Temperature Dependence of Lubricating Oils Using a Temperature-Scanning Technique (D 5133).

Gelation Index - Additional information from the SBT was generated with the concept of Gelation Index [*11*]. This came about in the late '80s as a response to the need to more clearly delineate among oils showing varying degrees of ogee formation -- a need to provide a numerical measurement of the level of gelation. The incremental derivative of the empirical MacCoull-Walther-Wright (MWW) viscosity-temperature equation was used.

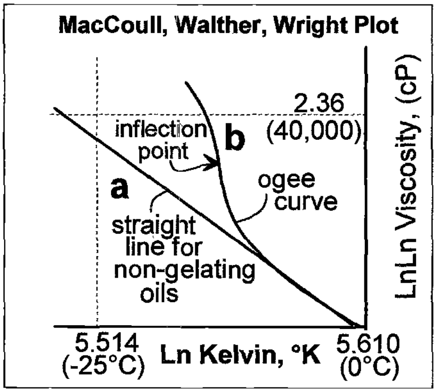


Figure 6

To show the concept, Figure 6 is a re-plot of the data of Figure 3 using the MWW equation. When this equation is applied to Oil **a**, a straight line is generated whose slope is related to the oil's change in viscosity with change in temperature.

When the MWW equation is applied to the gelated Oil **b** represented in Figure 3, only the higher temperature portion of the viscosity-temperature relationship unaffected by gelation gives a straight line. Development of gelation generates the now-familiar ogee curve is whose inflection point is indicated by an arrow. Now, if these two lines, one straight and one curved, are analyzed by taking the incremental values of their slopes (i.e. by manual differentiation), the resulting lines will look as in Figure 7.

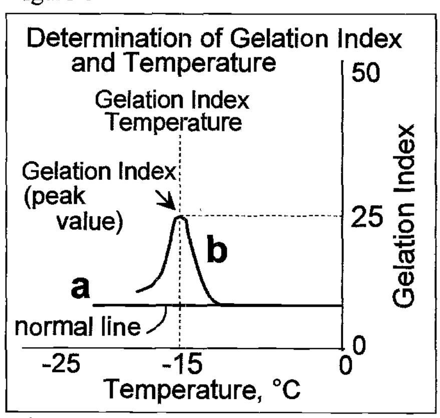


Figure 7

Figure 7 shows the incremental slopes of the curves of Figure 6 (see [*11*] for details). Non-gelating Oil **a** plots as a flat, straight line whose value is the slope of the MWW line. In contrast, gelating Oil **b** plots as curve showing a peak (note arrow) in the Gel Index curve which is the Gelation Index value, 25. The peak comes at the inflection point shown in Figure 6 and the temperature at which the Gelation Index occurs is called the Gelation Index Temperature, -15°C.

Precision of determining the Gelation Index was established in a round robin and in 1998 the technique and its precision was included in ASTM D 5133.

Hypothesis of Gel Formation and Torque Response in the SBT - In response to questions concerning the mechanism by which the SBT generates gelation information, an hypothesis is given in Appendix A.

Application of MRV TP-1 and SBT

North America

Specifications - Over the almost 20-year period since the Sioux Falls incident, the pumpability instruments developed in response have been used to monitor the acceptability of multigrade engine oils. Today both are applied in specifications around the world.

Discussion - Pumpability in the relatively cold climate of northern North America is a frequent winter concern and it would be expected that some single and multigrade oils would show problems. Since gelation of the engine oil is the most unpredictable form of pumpability problems, a study of the oils in North America for this response in bench tests was run on both the SBT and the MRV TP-1.

Permission to use the Institute of Materials engine oil database gave the opportunity of comparing the last seven years comprising 1750 samples from the market. The results are shown in Figure 8 for both instruments at a collection rate of 250 samples per year. (In the IOM data base, oils exhibiting yield stress in the MRV TP-1 were further analyzed for the level of yield stress and viscosity although that is not part of the D 4684 method.)

The data in Figure 8 show that, with the exception of 1992, more oils failed to meet the criteria of the SBT than the MRV TP-1. The author believes that this is related to the greater sensitivity of the SBT to the occurrence of gelation.

Several interesting facts may be noted. For example, the number of oils failing either MRV TP-1 or SBT decrease progressively from 1992 to 1995. It was in 1992 that ILSAC, the automobile manufacturers' international specification body began application

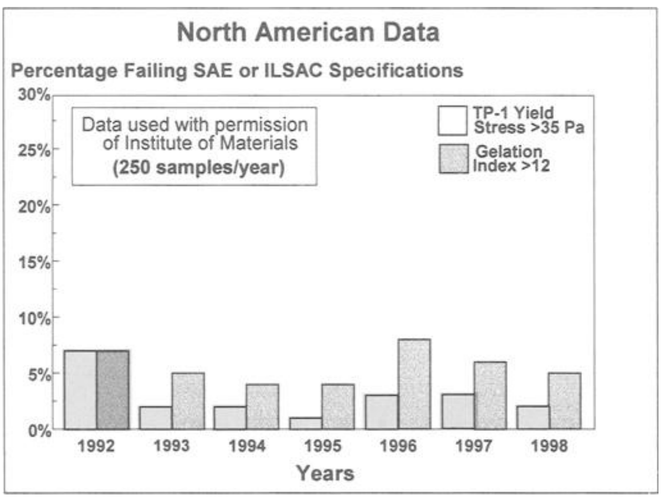


Figure 8

of ILSAC GF-1, the first specification from that group. By 1996, many more highly paraffinic base oils were being used to meet the requirements of ILSAC GF-2 and these oils required more care in pour-point depressant formulation to meet the requirement of having a Gelation Index of 12. This is evident in the rise in oils showing gelation.

Again, with time and experience, progressive improvement is seen. However, in general, the levels are higher in the 1996 to 1998 than in the period from 1992 to 1995 coinciding with the greater use of more highly paraffinic base oils. In almost every year, there were more oils showing failure with the SBT than the MRV TP-1 and this is again likely a reflection of the generally greater sensitivity of the SBT to gelation.

Overall, the data from North America show awareness and effort by engine oil manufacturers to improve the pumpability response of engine oils.

Europe

Specifications - European engine oils often include SAE Classification numbers and letters. ILSAC specifications are generally absent and, consequently, the requirement of meeting the Scanning Brookfield Gelation Index of 12 is not applied. In regard to need for pumpability control, much of Europe is moderate in temperature even during the winter (although the climates of the northern tier of countries and, particularly, Russia certainly see cold weather as demanding as that in North America). Figure 9 shows data similar to Figure 8.

Discussion - The data of Figure 9 indicate that, in general, control of pumpability-affecting phenomena is not a matter of as great a concern in Europe as it is in North America. Using SAE or ILSAC specifications (which may not be strictly required in Europe), double or triple the number of oils in North America are found to fail the criteria applied. Again, most of Europe may not need the protection as long as vehicles remain in the milder areas. However, for the colder portions of Europe such as the Scandinavian sector, oils with poor pumpability should certainly not be acceptable.

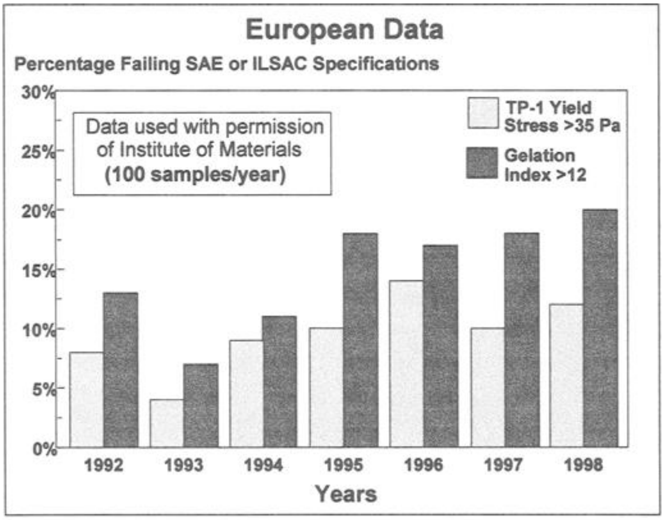


Figure 9

Second ASTM Pumpability Study

Startability/Pumpability Concerns

Since the first ASTM pumpability studies in the '70s previously discussed, one of the primary concerns has been clearly that if the engine will start, the oil must be able to be pumped. Consequently, a so-called "safety margin" of 5°C was established between the temperature of viscosity-limited startability and the lower Borderline Pumping Temperature based on the first pumpability studies.

The dramatic improvement in low-temperature engine startability over the last score years suggested that the engine might be able to be started with oils much more viscous than previous limits of 3200 to 4500 cP. Consequently, questions were raised by the SAE in 1992 regarding the startability and pumpability relationship and, in response, a new ASTM startability/pumpability study was begun. Interestingly, the SAE request also included a reevaluation of the bench instruments used to monitor the pumpability-related properties of engine oils at low-temperature since 1980.

A low-temperature engine performance (LTEP) task force was formed in 1992. Six years later, in 1998, work of that task force was ended and a formal report of results has since been made [*13*].

Startability - The first major focus of the LTEP study was to measure engine startability. To this author, the work by the participants was well done and revealing. It was shown that today's engines would start more readily and that the lower viscosity limits of the past were no longer operative. The work was then directed at establishing a new level of maximum viscosity for multigrade engine oils and this work is now in the hands of the SAE Engine Oil Viscosity Classification Task Force who have recently conducted a successful ballot establishing new viscosity levels. Publication of these levels will be made in the near future.

Pumpability, Viscosity-Limited - The first area of LTEP effort on pumpability was to identify the level of protection afforded by viscosity-limited oils. This was a reasonable beginning with newer engines perhaps capable of starting with oils having viscosities that couldn't be pumped. Four engines were used for the work which, in the author's opinion and from the experiences gained in the previous ASTM pumpability study, essentially limited the extension of conclusions that might be drawn to those four engines.

Another limitation was that the tests were carried only to the temperature considered sufficient to establish the safety margin for startability rather than establishing the Borderline Pumping Temperature used in the first ASTM pumpability study. Thus, it was not possible to later use the oils to build an instrumental correlation with the engines as had been done with the '70s pumpability reference oils shown in Figures 1, 2, and 5 and this, in turn, restricted meeting the request for evaluating the correlation of the bench instruments to these four engines.

The viscosity-limited pumpability work showed good correlation between a cold-room test with an exposed MRV and the older MRV D 3829 method. This was not surprising since, as shown in Figure 1, correlation of the MRV D 3829 with viscosity-limited oil has always been excellent.

Pumpability, Air-Binding - The most difficult part of the LTEP study -- the air-binding study -- was done last since proceeding earlier with this difficult yet critical part of the study might have taken up all of the funding and cold-room time available. Again, only four engines were available for this work. In the face of the first ASTM cold-room study showing the need for multiple engines to obtain relevant air-binding information from at least some of them, the challenge was apparent.

Unfortunately, no data were generated showing that the cold-room test programs used in the present study could produce subtle air-binding in a field-failing, 1980-vintage engine. The fact was that while some of the cold-room engine tests were successful with oils showing gelation in bench instruments, there was no evidence that this cold-room study was relevant to field conditions which had, years ago, shown the fallacy of believing too strongly in cold-room tests regarding gelation effects in the field.

The problem was compounded by the thought -- and potential misinterpretation by some -- that modern engines were now shown to be less sensitive to air-binding when, in fact, the LTEP test protocols themselves were untested for correspondence to the field. Moreover, engine builders stated that their engines were not redesigned for better response to gelation and, in some cases, might be more vulnerable as a consequence of changes in pump location, shallower oil sumps, and oil inlet tube and screen geometry. This technical controversy with its evident commercial overtones has been well covered in trade magazine articles in which the various views of those knowledgeable were presented [*13*].

Summary Comments on the LTEP Study

The LTEP program produced valuable information on startability and viscosity-limited pumpability. In regard to air-binding, the study can be interpreted in several ways. To some individuals the data seem to show that modern engines are less sensitive to gelation and, by implication, continued measurement of gelation tendencies at the level required by today's specifications are no longer necessary. The LTEP data indicate that a return to nearly the D 3928 level of yield stress is reasonable. On the other hand, others, including the engine manufacturers, have indicated that the design of the pump and pump inlet geometry has either not changed significantly or has become more vulnerable.

With the lack of critical cold-room proof of the test protocols and the statements of the OEMs, the author does not agree that LTEP data show that modern engines are less sensitive to air-binding. He believes that the data give hope but not reassurance since we have been this way before with harsh consequences. Nature is ultimately intransigent.

Rather, the author believes that protection from air-binding problems over the last 20 years is a direct result of the availability of bench tests known to correlate with field failures. As a consequence, the engine oils of North America provide much more protection against engine-damaging cold-weather conditions in comparison to central and southern Europe (in which control of low-temperature pumpability is not as pressing).

Summary of Author's Comments on the Overall Work Area

The last 30 or so years have seen much good work and high levels of cooperative activity to meet and overcome the challenges of man-made machines set in Nature's world-wide test cells. Pumpability has risen to become the dominant concern regarding the low-temperature performance of engine oils.

In studying the nature of pumpability and the rheological response of oils to cooling processes, much has been learned regarding the roles of base oils and additives in modifying and controlling adverse response of oils in the field. As a consequence, expanded use and convenience of the automobile and other forms of transportation has been garnered. Bench tests have been a source of much of this understanding.

Without doubt, there is much more to be learned and applied in regard to assuring dependable performance of engines at lower temperatures. With the experiences of the past, we should have considerably more knowledge and tools effective in that effort.

New Pumpability Challenges

Concerns Regarding the Pumpability of Used Engine Oils

Recent Developments Regarding Passenger Car Engine Oil - The combination of highly paraffinic base oils, need for careful selection of pour-point depressants, and the influence of the other additives has placed a premium on good pumpability measuring practices as noted earlier in this paper. However, the influence of yet another factor has recently arisen -- the influence of use of the engine oil on the pumpability of the oil and the additional impact of other additive chemistry introduced when the crankcase is topped up with a different engine oil. The latter factor was studied and reported in papers by Rhodes in '93 and '94 [*14,15*].

Essentially, it appears that with the development of more highly paraffinic base oils, the careful balance of the base oil, the pour-point depressant, the VI Improver, and the additive package must not only be obtained for the fresh oil but maintained in the used oil during its life in the engine. This of course brings in the effects of base stock and additive exposure to oxidation and, extending the above observations by Rhodes, the potential impact of oil admixtures between drain intervals. In turn, these effects must be coupled with the degree of oxidation and viscosity increase imposed by longer drain intervals.

Recent Developments Regarding Heavy Duty Diesel Engine Oil - Another impact on low-temperature pumpability has been encountered recently in the area of heavy duty diesel engines [*16*]. Government requirements for major reduction in the emission of oxides of nitrogen from these engines has resulted in retarding the combustion cycle with the result that large quantities of soot are generated. This soot rapidly loads the engine oil to levels of 10% and higher. The effect of such high soot loading on the action of dispersant additives and the strong possibility that lack of control of such soot levels could adversely affect pumpability even at higher ambient temperatures is a serious concern.

Response to Present and Future Pumpability Bench Test Needs

Extended Range Scanning Brookfield Technique -- Experimental Work

Recent papers on an extended viscosity range version of the Scanning Brookfield Technique showed that this approach was not only feasible but that even further extension could be considered [*17, 18*].

Consequently, a special method called the Scanning Brookfield Technique, Extended Range (SBT-XR) method was developed with particular Brookfield heads and analysis programs. The method has been recently applied to several engine oils included in the Institute of Materials Engine Oil Database. These oils were run in the SBT-XR mode in comparison to the standard SBT results used in present specifications and shown in the IOM Engine Oil Database. The maximum viscosity measurement using the SBT-XR is about 800,000 cP at 0.2 s^{-1} shear rate and the temperature range of the low temperature bath used was from +30° to -75°C.

Comparison of Fresh Passenger Car Engine Oils Using the SBT and SBT-XR Protocols

Well-Behaved Oils - The following data were gathered on a set of well-behaved (non-gelated) oils using the SBT-XR, the SBT, and the MRV TP-1 to determine how well the instruments agree when gelation is not present. These fresh oil tests were run primarily to determine the degree of correlation between the standard SBT and the SBT-XR.

Figure 10a shows the extended curve for Engine Oil K, a mineral-oil-based SAE 10W-40. The MRV TP-1 value obtained at -30°C is also shown as an open circle. The SBT-XR curve has the smoothly exponential shape associated with a well-behaved oil showing no gelation tendency. It will be noted that the value for the MRV TP-1 falls close to the SBT-XR curve as would be expected for simple, non-gelating oils.

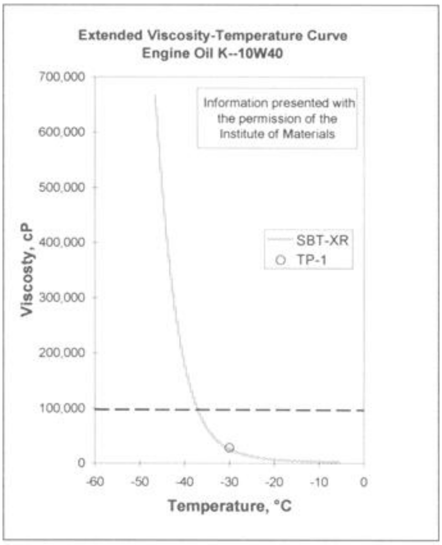


Figure 10a

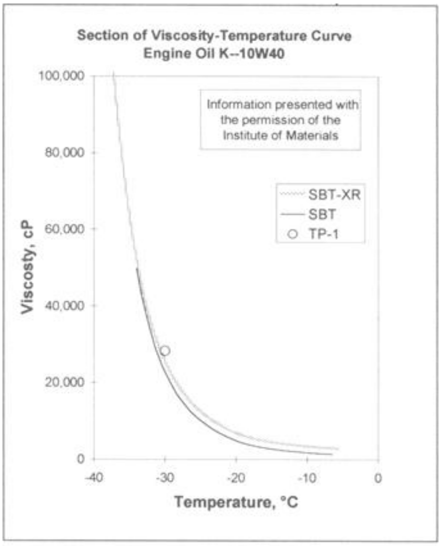


Figure 10b

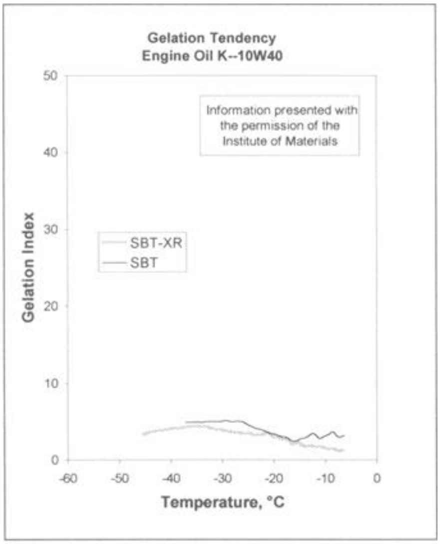


Figure 10c

A dashed horizontal line in Figure 10a is the upper limit for the expanded section shown in Figure 10b. Even with this magnified view, the data from the TBS-XR shows a smooth line. Moreover, the results of the standard range SBT data (obtained from the IOM database) shown in this latter figure, also falls in good agreement with the TBS-XR. Again, the MRV TP-1 value is plotted and falls in close agreement with both.

Figure 10c shows the associated Gelation Index curves with maximum values of from 4 to 5. The general forms of the curves are similar.

In general, the data of Figures 10a and 10b indicate that the curve generated by the extended-range SBT-XR fit encouragingly well with the normal range SBT.

Moreover, as would be expected for a well-behaved mineral oil, the SBT-XR, SBT, and MRV TP-1data agree well.

Engine Oil G -- Synthetic-Oil Based SAE 5W-50 - Comparison of the three instrumental modes of low-temperature viscometric analysis using a synthetic oil is shown in Figures 11a, b, and c on the following page.

Again, the plotted data in Figures 11a and 11b show that the SBT-XR and the MRV TP-1 values agree fairly well. Similarly, the more recent data obtained from the SBT-XR agree reasonably with the IOM SBT data published earlier.

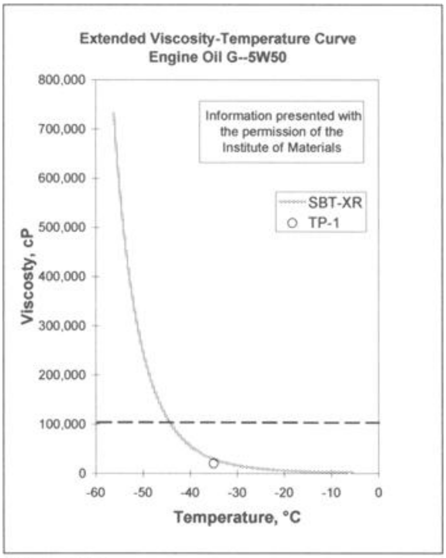


Figure 11a

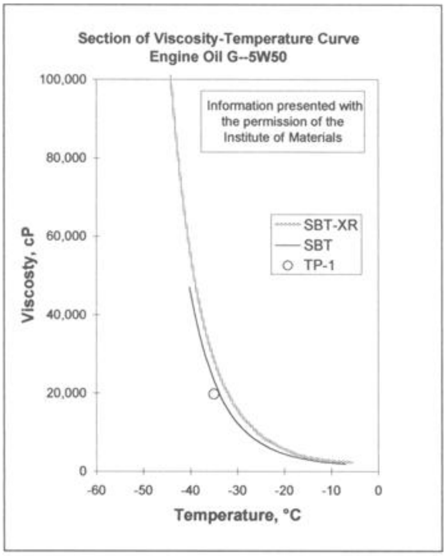


Figure 11b

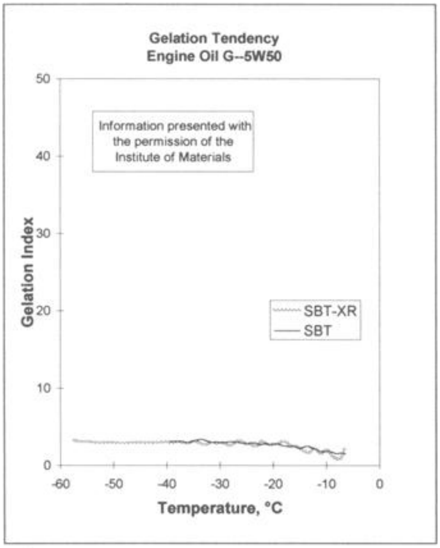


Figure 11c

The Gelation Index values for both the SBT-XR and the SBT are in good agreement and essentially horizontal over most of their range, thus showing no gelation tendency. The degree of agreement was considered to be even more promising considering the viscosity range over which this oil was measured. It would appear that the concept and application of the Gelation Index extends over a broad viscosity range.

Oils With Gelating Tendencies - At this point in the exploration of the application of the SBT-XR, it was appropriate to apply the technique to oils which had the capacity of forming gelation. Two SAE 15W-40 engine oils were chosen for this study, one of which was known from the IOM data base to have a significant Gelation Index of 37 and the other of which had a relatively low Gelation Index of 14.

Engine Oil A -- SAE 15W-40 (Higher Gelation Tendencies) -- The SBT-XR data on this oil produced the curve shown in Figure 12a. An evident ogee curve was generated as would be expected with an oil having significant gelation. In agreement with this information, the MRV TP-1 showed a yield stress of 175 and 210 Pascals in two tests. The viscosity is considerably lower than the value shown by the SBT-XR curve. However, the viscosities of the two tests on the MRV TP-1 agreed closely as is evident.

When the lower 100,000 cP section of Figure 12a is expanded as shown in Figure 12b and the SBT data added for comparison, it may be seen that the data from the SBT-XR and the SBT agreed closely -- to the point where the curves virtually lie on top of one another.

This agreement was further demonstrated by determining the Gelation Index obtained for Oil A from both the SBT-XR and the SBT. These curves are shown in Figure 12c. Again, it is evident that the agreement of the considerably lower torque SBT and the much higher torque SBT-XR is very close despite the difference in torque range. The Gelation Indexes and Gelation Index Temperatures are 36.5 @ -16.2°C and 37.9 @ -15.6°C for the SBT-XR and the SBT, respectively -- well within the precision given in ASTM Method D 5133.

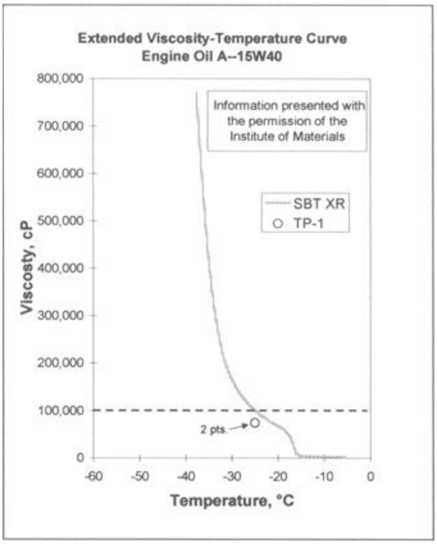


Figure 12a

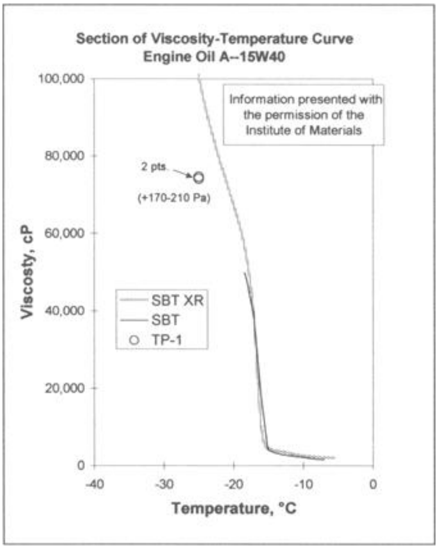


Figure 12b

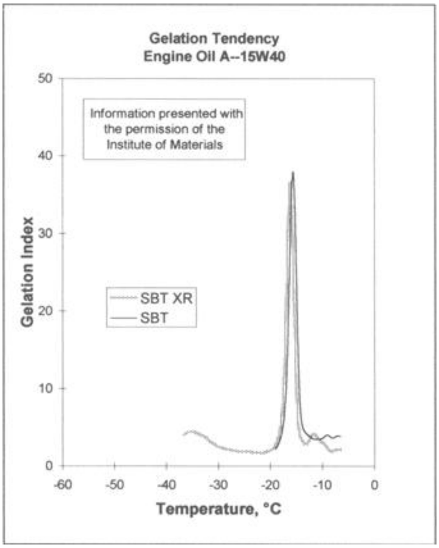


Figure 12c

Engine Oil D -- SAE 15W-40 -- (Lower Gelation Tendencies - It was of further interest to see what correlation would be found between the two Scanning Brookfield Technique protocols, SBT-XR and SBT, when used with an oil having a lower level of gelation. This comparison was particularly interesting considering the fact that the higher torque range of the SBT-XR method might reduce sensitivity to mild gelation.

It can be seen in Figure 13a that Oil D shows only a slight ogee in its curve. The remainder of the curve takes on the familiar exponential form. Data obtained on the MRV TP-1 is again displaced to lower values of viscosity.

When only the section of the curve below 100,000 cP is used, the ogee portion of the curve is made considerably more evident as shown in Figure 13b.

Regarding the level of agreement of the two different SBT protocols, Figures 13b and c show that agreement between the two SBT methods is surprisingly good. The higher level of torque applied in the SBT-XR seems to give equivalent sensitivity to gelation as the original SBT even at low gelation levels.

Comparing the numerical values obtained, the Gelation Indexes and Gelation Index Temperatures are 14.9 @ -14.1°C and 15.5 @ -13.9°C for the SBT-XR and the SBT methods, respectively -- again well within the precision of ASTM D 5133. With this information it seems apparent that the SBT-XR is applicable to a range of pumpability concerns.

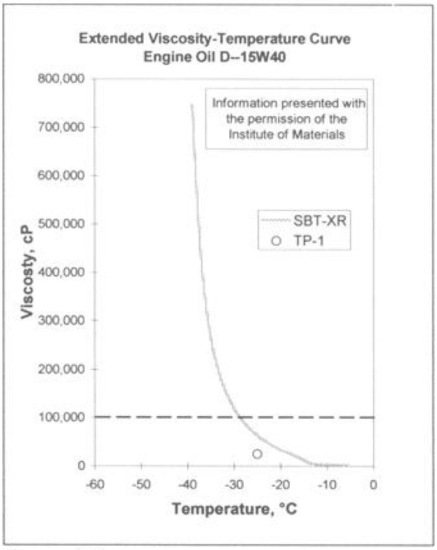


Figure 13a

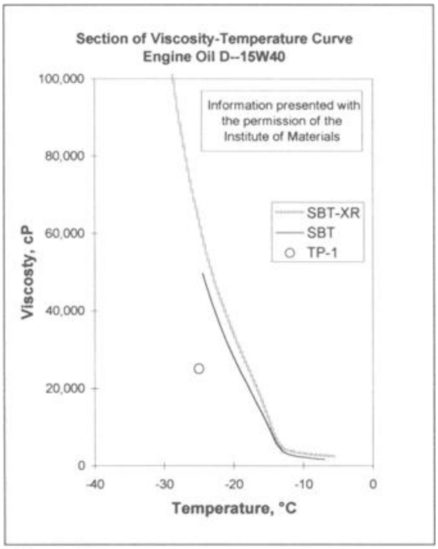


Figure 13b

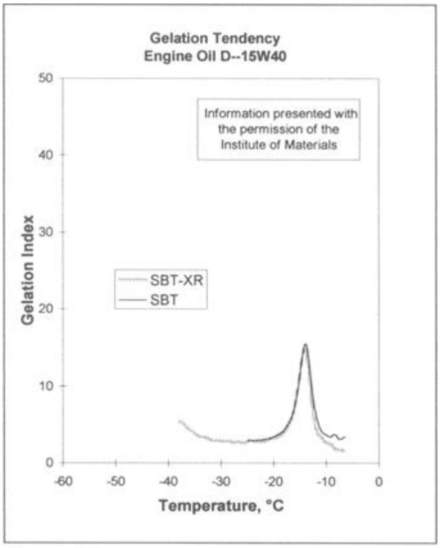


Figure 13c

The value of the MRV/TP-1 shown in Figures 13a and b is considerably lower than either the SBT-XR or even the original SBT. No yield stress was found by the TP-1 with this oil but at the low level of Gelation Index this was not surprising since yield stress is not often found below a Gelation Index of 25. This lack of correlation with mildly gelating engine oils supports the previous observation that the SBT, generally, is more sensitive to gelation. However, a few oils have been found in which the converse is true -- where the TP-1 is more sensitive.

Broad Application of the SBT-XR

It would appear from this new SBT work that although the experimental SBT-XR has a greater range of viscosity, little is lost regarding sensitivity to gelation. The high level of agreement between the SBT-XR and the normal range SBT is surprisingly since the use of the incremental derivative is a strong test of sensitivity and precision of the instrument.

With this data showing the range and precision of the SBT-XR, application to used oils seems important particularly with the rising level of concern about low-temperature pumpability of both oxidized passenger car engine oils and highly soot-laden heavy-duty engine oils.

Such subjects important to future questions of controlling engine oil pumpability are considered in the next section of this paper.

Modern Lubrication Pumpability Problems and Challenges

Sooted Oils and Pumpability Problems and Measurements

Background - In 1999 a government mandate to be implemented in 2002 regarding heavy-duty diesel emissions of NO_x has forced the industry to significantly retard engine timing. This has resulted in very high levels of soot in the engine oil as well as other consequences of such soot levels including valve train wear, shortened filter life, and (important from the considerations of this paper) loss of engine oil pumpability.

A recent well-developed and penetrating paper by McGeehan and associates documented these factors very clearly with clear warning about ignoring the significance of controlling soot [*16*].

Although comprising a relatively small part of the extensive work reported by McGeehan, the pumpability aspects and data related to these were considered by him as important to predicting proper engine function and protection. This was particularly the case considering the consequences of relatively poor pumpability at moderately low ambient temperatures.

Experimental Pumpability Findings Using the SBT-XR Protocol - On the basis of studies previously reported [*17*], the author's laboratory was requested to evaluate the rheological nature of these oils and this required use of the SBT-XR method. In particular, it required the method to be applied over a temperature range from 0° to -20°C or more with oils so sooted that viscosities could reach hundreds of thousands.

The data shown in Figures 14a, b, and c are taken from the paper by McGeehan, et al. and are striking in their rheological differences and the effect of dispersants on these properties. For example, Oils #1 and J are similar in viscosity at 0°C but very different thereafter with Oil J showing rheological characteristics suggesting either a viscosity-limited or air-binding flow problem for the engine shortly after the temperature reaches -5°C. The Gelation Index indicates a value of 16.

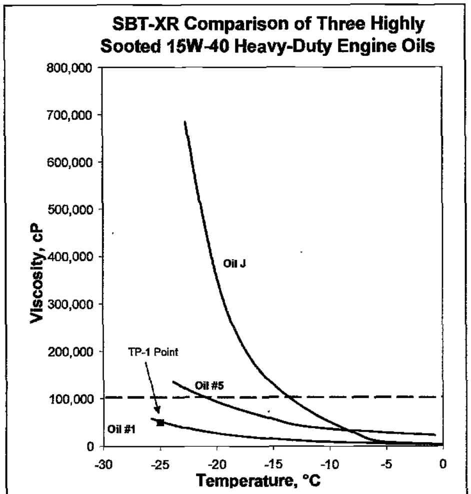


Figure 14a

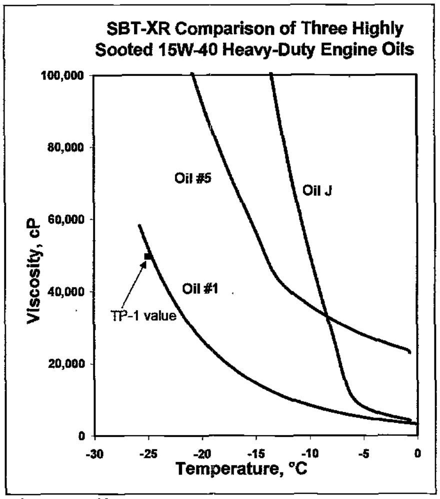


Figure 14b

Oil #5, on the other hand, starts at a relatively high viscosity of about 4500 cP at 0°C but from there becomes less viscous than Oil J although still not acceptable from the viewpoint of its comparatively high viscosity.

Oil J, with its evident Gelation Index at about -5°C also had the same magnitude Gelation Index when fresh at about -9°C. This suggests that the presence of high soot may move the Gelation Index Temperature to some higher temperature.

Oil #1 is exceptional among the three oils examined with the SBT-XR method in that it retained its exponential character (i.e. was free from evidence of gelation) and maintained the lowest viscosity of the three oils tested. It will also be noted that the TP-1 value of this oil fell on the SBT-XR curve, further indicating its freedom from gelation. In comparison, TP-1 values for Oils J and #5 were 108,000 and 93,000 cP, respectively.

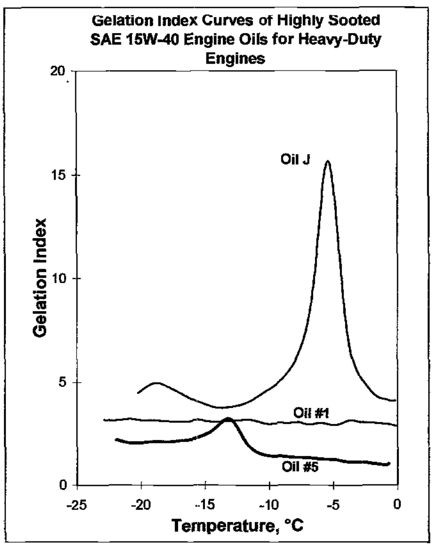


Figure 14c

Conclusions from the Sooted Engine Oil Tests

The results of this work on three sooted oils indicate that the use of the SBT-XR in characterizing the rheology of sooted engine oils at moderately low temperatures gives considerable information about the degree of dispersant control. With this bench test it was found that even at a level of 9% soot loading, it is possible for engine oils to retain acceptable control of pumpability at moderate and lower temperature as is evident from the bench data presented here and in McGeehan's original paper.

Oxidized Oils and Pumpability

Background - Today's emphasis on long oil-drain intervals has had the ancillary effect of de-emphasizing oil drains. In the case of leased cars, one of the consequences is that, with the low leakage rates associated with today's well-sealed engines, engine oils can be run for many thousands of miles without a warning light alerting the inattentive leasee that the oil should be changed.

It is not uncommon for 20,000 to 30,000 miles to be accumulated by the time the vehicle is drained and refilled. With smaller engines running at higher temperatures under greater operating loads, modern engine oils are placed under great duress.

One of the consequences of this extreme service is that the oil is so highly oxidized that it may experience pumpability problems at ambient temperatures as high as +5°C.

A small study was initiated to apply the SBT-XR method to determine the pumpability characteristics of oils having experienced over 20,000 miles without change.

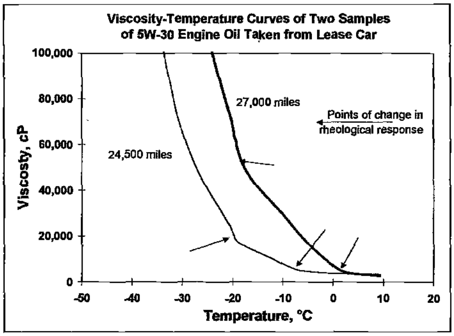


Figure 15

Results - Figure 15 shows both the results of generating well over 20,000 miles on an engine oil in relatively mild service and the rapid change in properties which occurs in only another 2500 miles.

Interestingly, there are several points on these curves showing the occurrence of some form of change in the rheology of the oil. This was analyzed further by generating the Gelation Index curves for both samples and these results are shown in Figure 16.

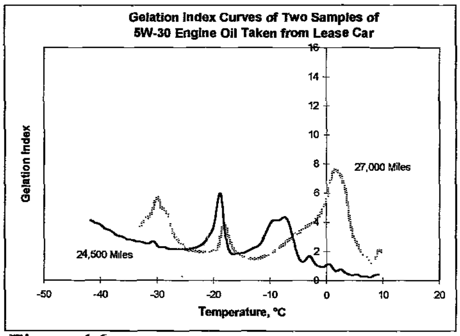


Figure 16

The Gelation Index plots of these oxidized oils show several areas of rheological change. In contrast to the singular Gelation Index peaks normally shown for fresh oils, multiple peaks are evident. Moreover, these peaks are found at relatively low levels of Gelation Index. This raised the question of the repeatability of such determinations.

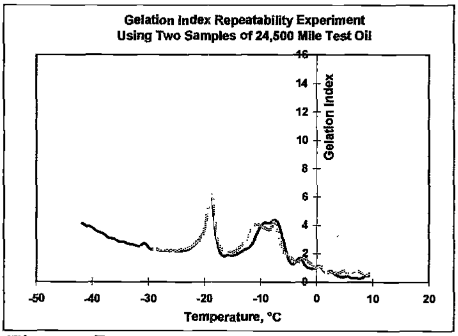


Figure 17

Accordingly, enough sample remained to run another analysis of the 24,500 mile oil sample and this is shown in Figure 17. The surprisingly close overlay of the two highly oxidized samples suggests that the Gelation Index approach to intramolecular and macromolecular association within a fluid may have much broader application particularly when applied to sooted and oxidized oils. These associations are evidently quite repeatable -- and likely to be indicative of oil condition.

Discussion and Conclusions

Imitation of Nature, Bench and Cold-Room Tests - The Past and Present

Imitation of Nature - Nature provides environments and conditions that are difficult to imitate and virtually impossible to predict. When incidents occur such as those of the winters of '80-'81 and '82-'83, the knowledge gained should be permanently embedded in technical consciousness -- together with the means of resolution and future control -- in order to prevent repetition.

Bench Tests - Resolution of the Sioux Falls incident was found using two bench tests showing good correlation with the field-failing engine oils. The tests were considered necessary in appraising the low-temperature behavior of oils and over the last 18 years no further epidemics have occurred. Over time and in the absence of epidemics, this apparent success led to speculation about whether modern engines might have become less susceptible to air-binding pumpability failures and, if so, led to the related question of whether the two bench tests continued to be relevant.

Consequently, the 1992 SAE request to ASTM for the LTEP cold-room study included a request to reassess the two bench instruments based on that study. To the author, meeting this latter request required 1) generating a test protocol proven capable of producing air-binding in the older field-failing engines, 2) applying this protocol to modern engines, and 3) assessing the applicability of the two instruments to modern engines.

Cold-Room Test Protocol Development - Cold-room test simulation of field conditions regarding startability are relatively straightforward as are tests of viscosity-limited pumpability. However, in regard to air-binding simulation of the field, the earlier ASTM cold-room studies [*1, 2, 4*] showed that a test protocol cannot be assumed correct and must be proven. The cold-room work of Stambaugh and O'Mara [*5*] stands as clear evidence of how much understanding and effort is necessary to imitate Nature.

LTEP Tests of Startability and Viscosity-Limited Pumpability - Cold-room information from the LTEP study provided good information on improved startability of modern engines. Although no information was generated providing the critical borderline pumpability temperatures for each of the test oils, the study did give pertinent information regarding appropriate relationships between startability and viscosity-limited pumpability values. This was perhaps the most important finding of the LTEP study and it set into motion activities by the SAE and others to change the SAE J300 Engine Oil Viscosity Classification System to reflect these findings.

LTEP Tests of Air-Binding Pumpability - Despite considerable efforts by the LTEP investigators with different protocols, no "bridge" to past field-failing oils and engines was generated. Without this bridge, the author feels that there is no basis by which to compare modern engines with those that had experienced field failure -- and that without this information, no evaluation of the relevance of the bench tests would seem possible.

It is salient that in those tests where the present LTEP study did show air-binding response, it was with oils having markedly higher yield stress levels. Specifically, the LTEP study found engine response at TP-1 yield stress levels of 70-105 Pa -- considerably higher than the maximum level of 35 Pa earlier shown [*8*] as necessary for prediction of the 1980-83 field-failing oils -- but very similar to the results of the first cold-room tests producing ASTM Method D 3928 and its maximum yield stress of 105 Pa.

Two interpretations of the LTEP air-binding studies are: 1) modern engines are less susceptible to air-binding; 2) the LTEP air-binding pumpability study is questionable and modern engines may be as susceptible to air-binding as before. If the latter is so, vigilance regarding air-binding using proven, field-correlated bench tests remains desirable.

The Present and Future - Development and Application of a New Pumpability Test for New and Used Oils

The Extended Range SBT - Present and future problems concern the pumpability of fresh lubricants at lower temperatures as well as used and soot-laden oils at relatively higher temperatures. Work has been shown in this paper on a new technique called the SBT-XR having both sensitivity to gelation and range in viscosity. In preliminary work with both simple and gelating fresh oils, the instrument and program gave surprisingly good agreement with the standard SBT results in both viscosity and Gelation Index and Gelation Index Temperatures. However, the SBT-XR instrumental approach was capable of reaching more than 700,000 cP.

Application to Highly Sooted, Heavy-Duty Engine Oils - Analysis of three highly soot-laden heavy-duty engine oils using the SBT-XR showed that these oils may or may not show the serious influence of soot in high concentrations. Pumpabilities of two of the three 15W-40 engine oils, Oils J and #5, were seriously compromised by the presence of soot. Both showed some level of gelation and viscosities ranged up into the hundreds of thousands of centiPoise at -25°C. In comparison, although carrying the same soot level, the third oil, Oil #1, showed essentially normal, exponential change of viscosity with temperature and stayed within the low-temperature grade for that oil.

It was also observed with Oil J that if the fresh oil initially showed the presence of gelation, the Gelation Index would remain the same with high levels of soot but would move to a higher Gelation Index Temperature.

Application to Highly Oxidized, Passenger Car Engine Oils - Increasing engine operating temperatures and extended oil drain intervals in leased vehicles have produced levels of oxidation in passenger car engine oils that readily exceed 100,000 cP at temperatures well above the low-temperature classification level. Moreover, the viscosity-temperature curves generated produce complex Gelation Index curves with several peaks. As mileage accumulates and viscosity increases, the peaks move up in temperature and peak height.

A repeatability study of one oil showed that as complex as these Gelation Index curve peaks were, they were as reproducible as the simple peaks associated with fresh oils. It would seem, then, that these peaks indicate some form of association of the molecular and particulate components of the oxidized oil.

Author's Observations

The last 40 years have seen major changes in the way lubricants are viewed. We come somewhat haltingly from a day long past in which all specifications for low temperatures came from values extrapolated from 100° and 210°F and engines and transmissions fared accordingly.

Over these years our technical efforts, generally, have been successful and in those cases in which we were not -- through either pride or ignorance (or both) -- Mother Nature furthered our education and our humility.

I believe we have done well to arrive at the end of a millennium with such an impressive group of automotive vehicles to serve us in all weather over most terrain with oils, fluids, and coolants sustaining these fine machines.

We still have a longer path to follow. Paraphrasing the words of others, the more we know, the more we know about what we don't know -- but the greater and quicker our ability to respond and transmute our ignorance into further knowledge.

Regarding the issues concerning pumpability, it may be that our problems with damaging air-binding oils are past. For myself, with the background we have generated regarding the juxtaposition of cold-room work and field experience, I find it difficult to extrapolate what we know "for sure" to confidence that air-binding is a past problem. Certainly, much strong and dedicated effort went into the last foray in cold-room testing and much understanding came out of it.

Concerns with the flow and pumpability of used oils, in general, and sooted oils and oxidized oils, in particular, are rapidly growing. Meeting these concerns will require new skills and instrumental approaches such as that presented in this paper. Task forces have already been formed in ASTM D02, Subcommittee 7 to address this area.

All in all, the latter half of this almost completed century has given us all a fair amount of technical exercise and entertainment -- may the next century be as clear in its challenges -- and may we use past experiences wisely and creatively to build a desirable future.

Acknowledgments

The author would like to express his gratitude for those who helped obtain the data presented in this paper -- in particular Larry Schember of the Savant Laboratories and Patrick O'Dell of the Tannas Co. Both worked with skill, diligence, and timeliness.

He is also very grateful to the reviewers of the paper and in particular to Dr. Harold Shaub, Editor of this ASTM STP 1388, for their dedicated efforts to help the author better express his information and concepts.

References

[1] *Low Temperature Lubricant Rheology Measurement and Relevance to Engine Operation,* ASTM STP 1143, Robert B. Rhodes, Ed., American Society for Testing and Materials, Conshohocken, Pennsylvania, 1992.

[2] Selby, T.W., "Viscosity and the Cranking Resistance of Engine Oils at Low Temperatures," Sixth World Petroleum Congress Proceedings, Section VI, Frankfurt, Germany, pp. 241-258, June 1963.

[3] *Low-Temperature Pumpability Characteristics of Engine Oils in Full-Scale Engines,* ASTM Data Series DS 57, 1975.

[4] Shaub, H., et al., "Predicting Low Temperature Engine Oil Pumpability with the MiniRotary Viscometer," SAE Paper #790732, SAE Passenger Car Meeting, Dearborn, Michigan, June 11-15, 1979.

[5] Stambaugh, R.L. and O'Mara, J.H., "Low Temperature Flow Properties of Engine Oils," SAE Paper #820509, SAE International Congress and Exposition, Detroit, Michigan, February 22-26, 1982.

[6] Stewart, R.M., et al., "Summary of ASTM Activities on Low Temperature Engine Oil Pumpability," SAE Paper #821206, SAE Fuels and Lubricants Meeting, Toronto, Canada, October 18-21, 1982.

[7] Smith, M.F., Jr., "Better Prediction of Engine Oil Pumpability Through a More Effective MRV Cooling Cycle," SAE Paper #831714, SAE Fuels and Lubricants Meeting, San Francisco, California, October 31-November 3, 1983.

[8] Henderson, K.O., et al., "New MiniRotary Viscometer Temperature Profiles That Predict Engine Oil Pumpability," SAE Paper #850443, SAE International Congress and Exposition, Detroit, Michigan, February 25 - March 23, 1985.

[9] Selby, T.W., The Scanning Brookfield Technique of Low-Temperature, Low-Shear Rheology - Its Inception, Development, and Applications," *Low-Temperature Lubricant Rheology Measurement and Relevance to Engine Operation,* ASTM STP 1143, Robert B. Rhodes, Ed., American Society for Testing and Materials, West Conshohocken, Pennsylvania, pp. 33-64, 1992.
[10] Selby, T.W., Written unpublished discussion of Reference [5] above. Included in STM RR-D02-1448 for ASTM Method D 5133, June 1999.
[11] Selby, T.W., "The Use of the Scanning Brookfield Technique to Study the Critical Degree of Gelation of Lubricants at Low Temperatures," SAE Paper #910746, SAE International Congress and Exposition, Detroit, Michigan, February 25 - March 1, 1991.
[12] ASTM, "Cold Starting and Pumpability Studies in Modern Engines," ASTM Research Report RR-D02-1442, American Society for Testing and Materials, West Conshohocken, November 1998.
[13] McFall, D., "The Weird World of Oil Gelation," April Issue, Vol. 5, #4, pp. 36-38, 1999, Lubes 'n Greases Magazine. "Weird Tales of Oil Gelation," May Issue, Vol. 5, #5, pp. 14-16, 18, 19, 1999
[14] Rhodes, R.B., "Low-Temperature Compatibility of Engine Lubricants and the Risk of Engine Pumpability Failure," SAE Technical Paper #932831, SAE International Fuels and Lubricants Meeting, October 1993.
[15] Rhodes, R.B., "Assessment of the Low-Temperature Incompatibility Risk of Commercial Engine Oils," *SAE 1994 Transactions, Journal of Fuels & Lubricants - Section 4,* Vol. 103, Society of Automotive Engineers, Warrendale, PA, 1995, pp. 1342-1351.
[16] McGeehan, J.A., et al., "The Pivotal Role of Crankcase Oil in Preventing Soot Wear and Extending Filter Life in Low Emission Diesel Engines," SAE Paper #1999-01-1525, International Spring Fuels and Lubricants Meeting and Exposition, Dearborn, Michigan, May 3-6 1999.
[17] Resio, S. and Selby, T.W., "The Critical Third Zone - A New, Fast Method of Measuring Engine Oil Pumpability at Low Temperatures from Pump to Lubrication Site," SAE Technical Paper #981411, SAE International Spring Fuels & Lubricants Meeting & Exposition, Dearborn, MI, 1998.
[18] Selby, T.W., "Recent Developments in Testing Lubricants," 6th International LFE Congress, Brussels, Belgium, June 2-4, 1999.

Appendix A

Sensitivity of the Scanning Brookfield Technique to oil systems which tend to cause air-binding encourages efforts to understand the phenomenon, particularly since the continuously turning rotor of the SBT would seem to inhibit the formation of structures in the oil. The following hypothesis would become more complex for sooted and oxidized oils:

The hypothesis proposed is, if an oil has a tendency to form an association of molecules -- a structural "network" -- at a given low temperature when in the quiescent state, when the oil is slowly cooled and sheared, this process will encourage the formation of such a network. Specifically, in contrast to the quiescent oil in which the network-building molecules must migrate slowly through the viscous medium, in the case of the Scanning Brookfield Technique the structure-building molecules are continuously supplied to the structure by slowly moving laminar oil flow (visualized by the differential motion of cylindrically concentric lamellae of oil under the impetus of the turning rotor).

As the slowly cooling oil passes through the critical temperature region(s), the network formation will proceed as rapidly as the network-building components are supplied. If there are relatively few of these components and are less frequently brought into position, the rate of network building will proceed more slowly. Moreover, since fewer components are available the extent of the network will be restricted even when all or most components are in place. In the case of the Scanning Brookfield Technique, it is easily seen that the rate and extent of network development is reflected by the Gelation Index. Such a network, of course, need not be rigid and, in fact, under the forces producing motion of the oil, the network would tend to become flexible and accomodate considerable distortion. Growth of the network would be expected to increase the resistance to motion of the bulk oil since motion of one portion of the network would "drag" other portions.

Although the motion of the oil on one hand is viewed as assisting the formation of the network, on the other hand, the force behind that same oil motion would tend to stress, deform, and perhaps rearrange the network. Thus, the overall process is viewed as a balance between the rate of component supply and the rate of network rearrangement under shearing conditions -- a process that reaches equilibrium when network building is in balance with network rearrangement.

Figure A shows a simple diagram of the laminar representation of this concept with an effort to express the overall interactive thickening of the whole fluid by showing contiguous layers of moving fluid developing a network.

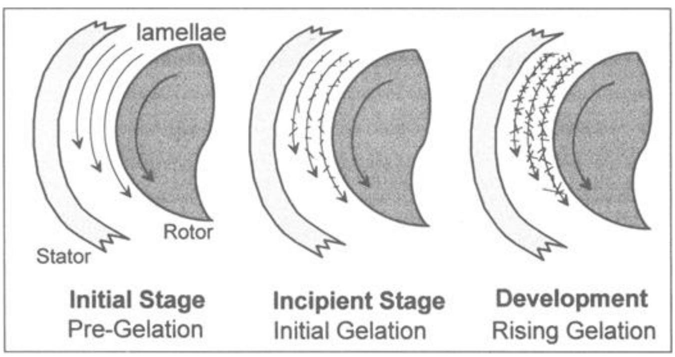


Figure A - Vertical view of rotor/stator, "lamellae", and network growth.

Fred W. Girshick,[1] Lewis H. Gaines,[2] and David J. Martella[3]

Designing Low Temperature Performance for Diverse Basestocks

Reference: Girshick, F. W., Gaines, L. H., and Martella, D. J., "**Designing Low Temperature Performance for Diverse Basestocks,**" *Oil Flow Studies at Low Temperatures in Modern Engines, ASTM STP 1388,* H. Shaub, Ed., American Society for Testing and Materials, West Conshohocken, PA, 2000.

Abstract

A new class of Lubricant Oil Flow Improvers (LOFI) was developed using statistically-based molecular design techniques, employing model compound studies and fundamental mechanistic understanding. These LOFI's were designed to be effective in a broad range of solvent- and catalytically-dewaxed basestocks, including API Group II and III hydrocracked stocks.

Basestocks for crankcase oil formulations are changing in composition. The volume of hydrocracked basestocks is increasing, realtive to solvent extracted basestocks. New dewaxing processes have been successfully introduced, such as catalytic dewaxing and catalytic iso-dewaxing. These process changes, and increasingly varied crude sources, lead to significantly different types and distributions of wax in the basestock.

LOFI'S, also called pour point depressants, optimized for traditional solvent-extracted, solvent-dewaxed basestocks may not work efficiently in these newer stocks, or may not work at all. Conversely, LOFI's designed for the newer basestocks may be ineffective in the more traditional basestocks which still dominate the market. An ideal LOFI will be able to treat both types of basestock with a wide range of viscosity modifiers, to maximize formulators flexibility.

The traditional empirical approaches to selecting LOFI can require excessive time and test costs as the basestock slate becomes more complicated and new test requirements are introduced. The design and selection approach presented here is based on the use of model diagnostic compounds which maximize the response of oil formulations to the

---

[1] Heavy Duty Engine Oil Formulation Leader, Infineum USA L.P., Linden, NJ 07036
[2] North American Market Manager for Small Engines and Marine, Infineum USA L.P., Linden, NJ 07036
[3] Chemist, Infineum USA L.P., Linden, NJ 07036

molecular parameters of the LOFI. These parameters include molecular weight, average carbon number of the side chains, and the distribution of side chain lengths.

Keywords:

Basestock, Flow Improver, LOFI, MRV, Pour Point, PPD, TP-1

Background

The historically straightforward process of insuring adequate low temperature performance for crankcase oils has become much more complex. Changes in both basestocks and performance test requirements are causing oil formulators to seek ways to optimize basestock costs, to simply formulation recipes, and to deliver reliable performance against increasingly stringent low temperature performance requirements. Properly selected pour point depressants can allow formulators to take advantage of these new basestock options while simultaneously minimizing overall grade slate complexity and assuring robust product performance under all temperature environments.

Crankcase Oil Product Slate Complexity is Increasing

Accelerating this trend is the general use of a greater variety of basestocks based on combinations of hydrocracking and catalytic dewaxing technologies. These approaches, giving greater product flexibility and improved economics for the basestock refiners and improved formulation options for oil marketers, are in place worldwide. These alternatives are expected to show increasing penetration against the traditional crude distillation and solvent dewaxed production routes. In North America, catalytically dewaxed basestocks now represent more than 20% of available capacity. Multiple plants are now operating in Asia with world penetration expected to approach 40% by 2005[4].

As basestock options have grown, so have the low temperature performance requirements become more stringent. For most oils, the long-favored ASTM Test Method D 97 for Pour Point of Petroleum Products has been supplemented by ASTM Test Method D 4684 for Determination of Yield Stress and Apparent Viscosity of Engine Oils at Low Temperature (MRV TP-1) and ASTM Test Method D 5133 for Low Temperature, Low Shear Rate, Viscosity/Temperature Dependence of Lubricating Oils Using a Temperature-Scanning Technique (Scanning Brookfield). The MRV TP-1 test was developed to prevent field pumpability failures. Field failures occurred under a specific low temperature profile resulting in multiple engine failures in the Northern

[4] Harts Lubricant World, November 1998

United States. The Scanning Brookfield (Gelation Index) Test, designed to be sensitive to the first stages of wax formation, was introduced to supplement the MRV TP-1 test. A brief summary of key low temperature tests is shown in Table 1.

During this study, it was found that MRV TP-1 requirements were more stringent than those of Scanning Brookfield. That is, all the oils that passed MRV TP-1 also passed Scanning Brookfield. For that reason, only MRV TP-1 results are shown below.

Table 1 - Key Low Temperature Tests for Defining Oil Formulations

• **Cold Cranking Simulator** Excellent correlation with low temperature engine behavior	• **Scanning Brookfield** Slow cooling cycle Derivative of viscosity measurement sensitive to initial wax formation
• **MRV TP-1** Excellent correlation with low temperature engine pumpability and field experience Slow cooling cycle	• **Pour Point** Rapid Cooling Used for product QC control

Formulators may also choose among a range of Viscosity Modifier (VM) options including olefin copolymers, polymethacrylates, and hydrogenated styrene-isoprene copolymers. Paralleling these component and test changes is a growing awareness of the potential interactions among all oil components including the dispersant inhibitor (DI) package.

Lube Oil Flow Improvers Can Improve Oil Performance

By using an appropriately selected Lube Oil Flow Improver (LOFI), robust formulations can be developed which will meet or exceed the typical test standards. However, as formulation complexity and options have increased, the selection of LOFI products has become more complicated. Blenders will likely be handling a variety of basestock types and will be considering several DI/VM combinations for their total range of crankcase oils. As a result they will have to search harder for a single LOFI solution applicable across the maximum number of formulated products.

Table 2 demonstrates the flexibility that appropriately selected LOFIs can offer the formulator. While VM and basestock selection dominate cold-cranking performance, LOFIs can deliver the other required performance levels. However, practical attainment of this benefit requires: (1) rapid identification of the correct LOFI for each individual oil, and (2) broadly applicable LOFI's to avoid the need for multiple LOFI grades at plants handling a range of basestock types and viscosity grades.

Table 2 - Influence of Crankcase Oil Components on Low Temperature Performance

Test Procedure	Basestock Impact	VM Impact	PPD Impact	Comments
Cold Cranking	Major	Major	Minor	High shear test with good field correlation
Pour Point	Major	Moderate	Major	Frequent QC testing focuses ongoing attention
MRV TP-1	Major	Moderate	Major	Strong field correlation. Large "safety margin" vs spec is often achievable
Scanning Brookfield	Major	Moderate	Major	Small safety margin. Poor reproducibility and repeatability

The Molecular Basis of LOFI Performance

The objective of this paper is to develop tools which will deliver customer solutions quickly. By narrowing the LOFI search range, time and test costs are reduced. In addition, by understanding the differences in response between classes of basestock, we are able to confirm and understand the occasional need to handle different LOFI's for different viscosity grades.

LOFI polymers work by co-crystallizing with petroleum wax and constraining crystal growth to a large number of relatively small crystals with a minimum of three-dimensional structure. Such crystals are less prone to agglomeration and entanglement, either of which will reduce oil flow at low temperatures.

Strongly performing LOFIs are comb polymers, *i.e.*, linear polymers containing "long" side-chains. The major classes of the LOFI comb polymers are polymethacrylates, styrene-maleate copolymers, α-olefin-maleate copolymers and fumarate vinyl acetate (FVA) copolymers. For cases where the LOFI is desired as part of the VM (a pour depressed VM), LOFI solubility in the VM becomes critical and not all classes of LOFI and VM are compatible.

LOFI Design Criteria

One LOFI chemistry is based on fumarate-vinyl acetate (FVA) copolymers. The general structure of FVA polymer is shown below in Figure 1. While this illustration is based on fumarate vinyl acetate polymers, the same general design criteria and flexibility applies to the other LOFI polymers as well.

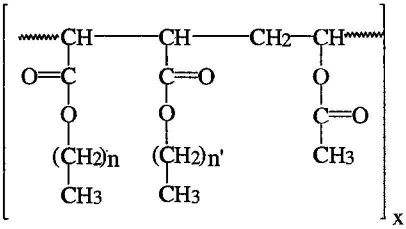


Figure 1

LOFI polymers can be engineered with three degrees of freedom. Of primary importance is the average side chain carbon number (average of n and n'). The average carbon number determines the oil solubility of the LOFI. The first crystals of wax which form when an untreated lubricating oil cools are generally very large and plate-like. In determining the optimum side-chain carbon number, the objective is for the LOFI polymer to co-crystallize with the initial wax crystals and modify their shape and thereby prevent structure formation. The second degree of freedom is the molecular weight (x) of the copolymer. The molecular weight of the copolymer helps to control the shape of the growing wax crystal as more wax precipitates on cooling. Finally the carbon number distribution (range of n and n') influences how different segments of the LOFI copolymer interact with the wax crystals.

By careful design, we have synthesized a diagnostic set of FVA LOFIs that allows us to determine the optimum position on this three-dimensional space of average side-chain carbon number, polymer molecular weight, and carbon number distribution. This set of diagnostic FVA LOFIs is used to identify the proper LOFI for a particular basestock. This minimizes both time and test costs and provides the methodology for designing new FVA LOFIs. The following three examples demonstrate the methodology. The first example is a solvent-dewaxed API Group I basestock. The second example is a hydrocracked, solvent-dewaxed basestock, API Group II basestock, and the third example is a catalytically-dewaxed basestock API Group II basestock.

Example 1: Solvent Dewaxed Group 1 Basestock

The first example is an SAE 5W-30 lubricant formulated with a solvent-dewaxed Group I basestock in the first step of the LOFI selection process: the diagnostic FVA LOFIs. Figure 2 shows that the optimum side-chain carbon number is slightly lower than the middle of the range. At this side-chain carbon number, the LOFI polymer has just the right solubility to co-crystallize with the initial wax precipitating from the basestock as it is cooled. LOFI polymers with higher side-chain carbon numbers precipitate before the initial wax and have little effect on the low temperature performance of the basestock. LOFI polymers with side-chain carbon numbers lower than the optimum precipitate with the bulk of the basestock wax and degrade with low-temperature performance.

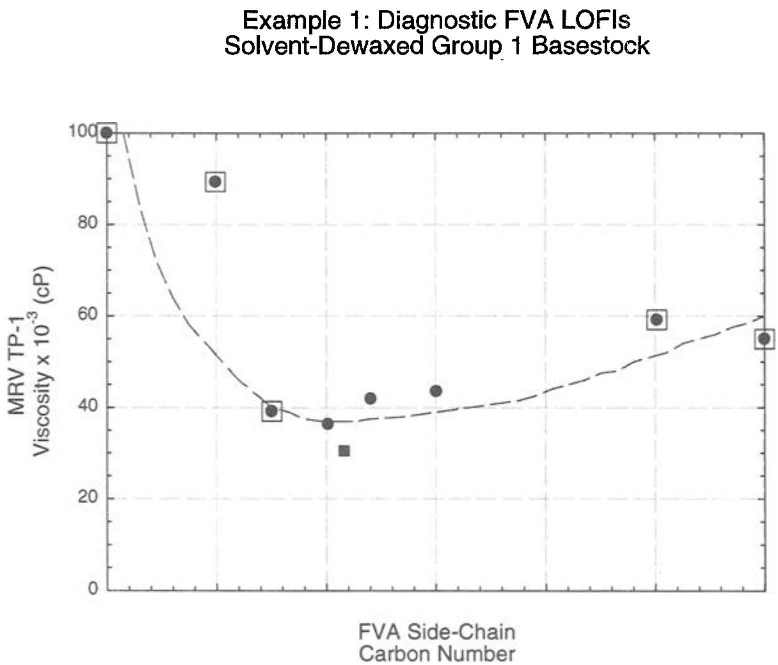


Figure 2

The second step in LOFI selection is to evaluate the response of the basestock to the molecular weight of the LOFI polymer. The diagnostic LOFIs have been designed as a matched set, pairing a low molecular weight and high molecular weight version of each.

The matched pairs are then screened around the optimum carbon number. In Figure 3, there is a slight preference for the low molecular weight LOFI polymers.

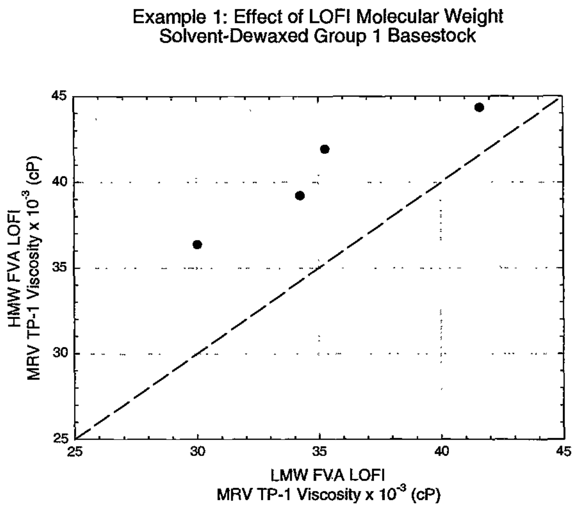


Figure 3

The third step of LOFI selection is to optimize the side-chain carbon number distribution. There are many possible combinations of side-chains for a given average sice-chain number. These combinations can be tailored to optimize how different sections of the polymer interact with later stages of wax precipitation. However, because the low-temperature performance of the diagnostic LOFIs is good, a simple distribution is sufficient in this case.

Thus, for this basestock the optimum FVA should have a low-mid average side-chain carbon number, a low molecular weight, and a simple distribution. As shown in Figure 2, LOFI "A" fulfills these requirements and gives excellent low-temperature performance in this basestock.

Example 2: Solvent-Dewaxed Group 2 Basestock

The second example is an SAE 15W-40 lubricant formulated with a hydrocracked, solvent-dewaxed Group II basestock. The diagnostic FVA LOFIs were screened in this formulation, as shown in Figure 4. The optimum side-chain carbon number is again in the middle of the diagnostic set. Figure 5 shows that there is no molecular response for the FVA LOFI. The excellent low-temperature response of the diagnostic LOFIs suggests that a simple side-chain distribution will be sufficient for this basestock. Thus, for this particular basestock the optimum FVA should have a mid-average side-chain carbon number, low molecular weight, and a simple distribution. As shown in Figure 4, LOFI "B" fulfills these requirements and give excellent low-temperature performance in this basestock.

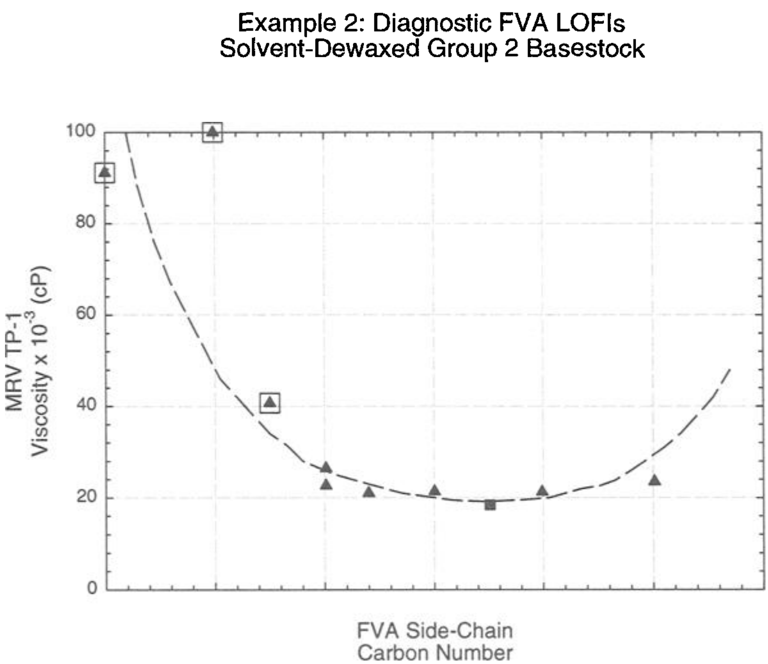


Figure 4

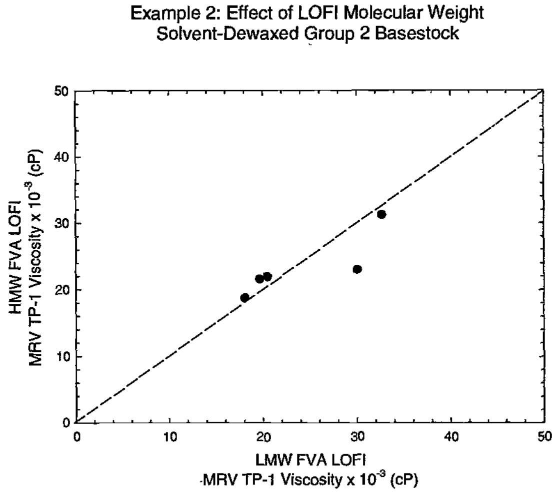


Figure 5

Example 3: Catalytically Dewaxed Group 2 Basestock

The third example is an SAE 10W-40 lubricant formulated with a hydrocracked, catalytically dewaxed Group II basestock. The diagnostic FVA LOFIs (Figure 6), show that the optimum side-chain carbon number is slightly higher than the middle of the range. The molecular response is shown in Figure 7. Unlike the solvent dewaxed basestocks, these results show that there is a clear preference for the high molecular weight FVA LOFIs. The good performance of the diagnostic FVA LOFIs again suggests that the simple side-chain distribution will be sufficient for this basestock. Thus, for this particular basestock, the optimum FVA should have mid-average side-chain carbon number, high molecular weight, and a simple distribution. As shown in Figure 6, LOFI "C" fulfills these requirements and give excellent low-temperature performance in this basestock.

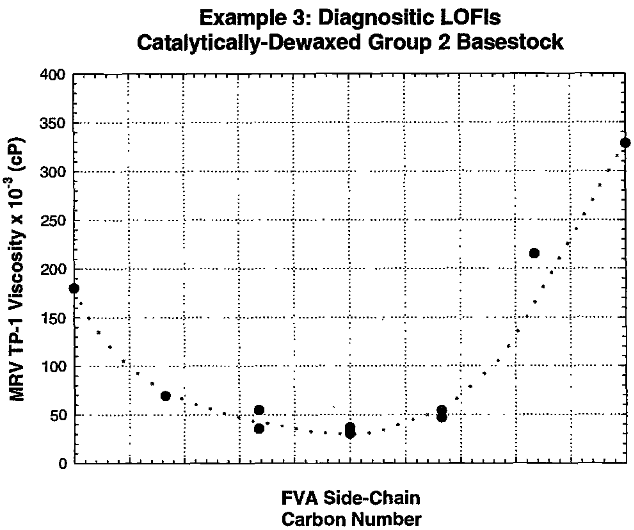


Figure 6

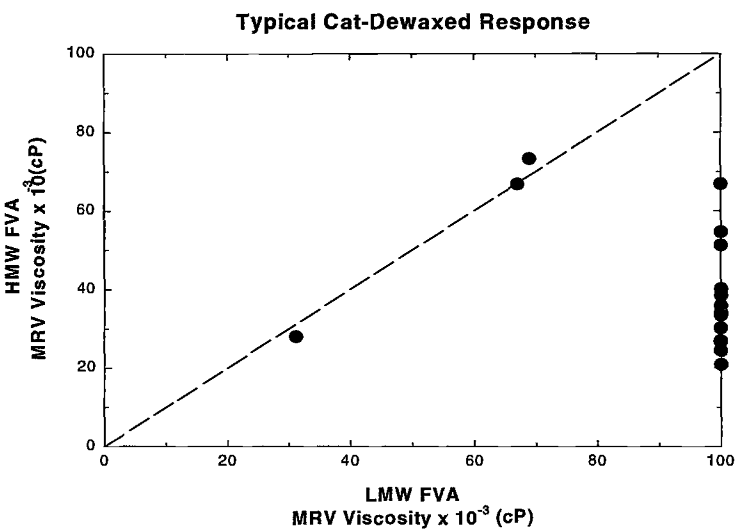


Figure 7

Conclusions

Basestock manufacturing techniques and resultant low temperature properties are changing rapidly. Robust flow improvers for use with a diverse basestock slate are needed, but development costs must be controlled. Molecular modelling, coupled with statistically-designed model compound syntheses, is the most effective route towards new additive development and selection.

Richard M. Webber,[1] Herman F. George,[2] and Michael J. Covitch[3]

The Relation between Low-Temperature Rheology of Lubricating Mineral Oils and Gelatin Index

---

Reference: Webber, R. M., George, H. F., and Covitch, M. J., **"The Relation between Low-Temperature Rheology of Lubricating Mineral Oils and Gelation Index,"** *Oil Flow Studies at Low Temperatures in Modern Engines, ASTM STP 1388*, H. Shaub, Ed., American Society for Testing and Materials, West Conshohocken, PA, 2000.

Abstract: The association of the predominantly *n*-paraffinic components of mineral oils into wax nuclei that grow and interact to form macroscopic crystalline structures leads to highly non-Newtonian rheology that is strongly dependent on both temperature and stress history. Upon cooling to temperatures lower than the onset temperature for wax crystallization (T_c), the viscosity-temperature curve shows a high activation energy region that persists over a narrow temperature range (~3-5 °C). In this temperature range, the oil transitions from a homogenous solution to a two-phase wax crystallite dispersion. By applying controlled stress rheometric techniques, we show that the high activation region is related to gelation index described in ASTM D 5133, and that the gelation index temperature corresponds to T_c. The high activation energy region is associated with the relief of supersaturation accrued within the oil upon cooling to temperatures below that of the saturation temperatures of the paraffin molecules in solution. Therefore, the gelation index characterizes the onset of nucleation rather than the formation of macroscopic wax crystal structures that would be associated with a yield stress and gelation. The activation energy and rheology at $T < T_c$ depend on temperature and stress history, the paraffin molecular weight distribution in the oil, and the concentration of pour-point depressant. We show that the gelation index parameter is non-unique, coupled to both the effects of stress history on viscosity and the dependence of nucleation on the *n*-paraffin molecular weight distribution. It is not surprising then that no correlation between gelation index and MRV TP-1 yield stress (ASTM D 4684) is observed.

Keywords: gelation index, nucleation, stress history, yield stress, crystallization, rheology, wax

---

[1]Research engineer, [2]senior engineer, and [3]senior research scientist, The Lubrizol Corporation, 29400 Lakeland Boulevard, Wickliffe, OH 44092.

Introduction

Lubricant viscosity is a critical parameter used by automotive engineers in the design of bearings, valve train components, ring belt hardware, gear assemblies and transmission systems. The ultimate goal is that sufficiently thick oil films are provided to ensure equipment durability, while recognizing that minimizing viscosity is desired for enhanced fuel economy. The lubrication process involves supply, entrainment and discharge of oil from the tribological contact. The physical properties of engine oils are such that the supply and discharge processes are quite facile under normal conditions.

When engines are started at low temperatures, however, oil supply can be impeded by high viscosity and, in some cases, fluid gelation in the oil pan, phenomena resulting primarily from crystallization of predominantly *n*-paraffinic components within the oil. Furthermore, if oil viscosity in the bearings is too high, the engine might not be capable of starting. This is the reason the Society of Automotive Engineers (SAE) incorporated several measures of low-temperature viscosity and gelation in the Engine Oil Viscosity Classification standard SAE J300. The Cold Cranking Simulator test (CCS), Test Method for Apparent Viscosity of Engine Oils Between –30 and –5 °C Using the Cold-Cranking Simulator (ASTM D 5293), measures the viscosity of oils under simulated starting conditions (high shear rates); whereas the Mini Rotary Viscometer test (MRV), Test Method for Determination of Yield Stress and Apparent Viscosity of Engine Oils at Low Temperature (ASTM D 4684), relates to the ability of the oil to flow to the pump so that it can be pumped to the engine. The latter test measures both yield stress and low shear-rate viscosity after the lubricant has been slowly cooled to the test temperature following a well-defined cooling cycle labeled TP-1 [*1*]. Yield stress is a measure of the tendency of the oil to resist flow due to gelation. Low shear-rate viscosity is a measure of the pumpability of the oil. Both conditions, for example a large yield stress or a high viscosity, may lead to engine failure because of inadequate lubricant supply. The former is often associated with a failure mechanism called air-binding, that is the uptake of air into the oil pump rather than lubricant; the later is commonly associated with a mechanism of flow-limited failure, that is the inability to provide enough lubricant to the engine in a timely manner [*2*]. The CCS and MRV limits in SAE J300 have historically been based upon cold-room testing of gasoline engines, supported by a consortium of engine builders, oil and additive companies.

Another test proposed to measure oil gelation at low temperatures is the Scanning Brookfield test (SBT), Test Method for Low Temperature, Low Shear Rate, Viscosity/Temperature Dependence of Lubricating Oils Using a Temperature Scanning Technique (ASTM D 5133); its use has been described by Selby [*3-5*]. This viscometer measures viscosity continuously at constant shear rate as the oil is cooled slowly from -5 to -45 °C at a constant rate of 1 °C/h. It was observed that the MacCoull/Walther/Wright (M/W/W) plot of viscosity (η) as a function of temperature (T) (plotted as $loglog\eta$ versus logT) was not always linear over the entire low-temperature range. Some oils exhibited an abrupt increase in viscosity over a narrow temperature range, and the phenomenon was found to be fairly reproducible. Selby proposed that the strength of the viscosity increase, specifically the maximum slope of the M/W/W viscosity-temperature curve, positively correlates with gelation within the oil; he coined the term "gelation index" (G_i^{SB}),

$$G_i^{SB} \equiv -\frac{d\,loglog(\eta)}{d\,log\mathrm{T}}\bigg|_{max} \quad (1)$$

where η is the viscosity in centipoise, T the temperature in Kelvin, and the superscript SB designates gelation index determined according to D 5133. This definition in effect equates the concept of gelation with the abrupt viscosity increase and, therefore, the onset of a yield stress, the later because gelation implies a transition from viscous to predominantly elastic behavior. A macroscopic wax crystal network or structure therefore must form in the oil. Achieving flow requires breaking this structure, and the stress required to break the structure is the yield stress.

A number of engine manufacturers have embraced the gelation index as a means of providing an added, that is in addition to D 4684, measure of protection against air-binding engine failures in the field. Data from an industry-wide study of the 1980-81 Sioux Falls incident using Pumpability Reference Oils (PRO) is often cited as technical justification for incorporating G_i^{SB} in various OEM factory fill and engine oil performance category specifications such as ILSAC GF-2 and GF-3. A summary of this PRO data [*1,6*] is included as Table 1. Close examination of this data, however, reveals that the apparent failure of the MRV TP-1 test to identify certain air-binding oils (such as PRO 1, 10 and 13) was simply due to the fact that the engine borderline pumping temperature (T^{BP}) was lower than the MRV TP-1 test temperature. We recently conducted MRV TP-1 tests on PRO 10 and 13 at temperatures just above and below the reported T^{BP} and found that the MRV TP-1 correctly identified PRO 10 as an air-binding oil between –30 and –35 °C and PRO 13 as a flow-limited failing oil in the same temperature regime (note that the critical MRV TP-1 viscosity for late 1970 model passenger car engines was 30,000 cP so this oil would have failed at –35 °C under those standards). Therefore, including a G_i^{SB} standard does not appear to add value beyond the MRV TP-1 standard. An important question, however, remains, what aspect of oil performance is characterized by measuring gelation index?

As pointed out by Kinker et al. [*6*], although the lubricant is undisturbed during cooling in both engines and the MRV TP-1 test, the SBT imposes the "artificial stimulus of the [continuously] turning spindle." The authors demonstrate that the SBT and MRV TP-1 viscometers agree well in the absence of stirring (i.e., when the SBT spindle is stationary during cooling), but do not agree when the spindle is continuously rotating as the oil is cooled. They conclude "the MRV TP-1 and the SBT often measure different rheological characteristics of engine oils." For 21 European multigrade engine oils, Kinker et al. did not observe a correlation between G_i^{SB} values and yield stress measured in the MRV TP-1 test.

The ASTM Low Temperature Engine Performance task force was commissioned in 1992 to "assess [among other things] the benefits and limitations of current methods for identifying oils that could result in pumpability failures in engines." One of its specific goals was to determine if gelation index adds value to the pumpability protection already provided by the MRV TP-1 test. As amply demonstrated in other papers presented at this Symposium, the LTEP team could not induce air-binding failure in high G_i^{SB} oils that passed the MRV TP-1 test except by significantly lowering sump volume. A more limited low temperature engine pumpability study on modern SAE 5W-30 oils reported by Koenitzer et al. [*7*] also failed to show added value for the gelation index measure.

Table 1 - *PRO pumpability data from engine and bench tests [1,6]. Underlined values were measured by the authors. SAE viscosity grades (W) are indicated for each sample.*

PRO	G_i^{SB} a)	T^{Gi} (°C) a)	σ_o^M (Pa) b)	η^M (cP) b)	T^M (°C) b)	T^{BP} (°C) c)	Failure Mode d)
1(5W)	8.7	-21	pass	pass	-30	-36	AB
9(10W)	8.6	-19	fail	---	-25	-26.5	AB
10(5W)	>22.8	-28	pass	pass, 10,100	-30	-32.5	AB
	17.2	-27	fail	---	-35	---	---
13(10W)	13.1	-22	pass	pass	-25	-32	FL
	---	---	pass	16,000	-30	---	---
	---	---	pass	56,700	-35	---	---
30(10W)	37.1	-12	ND e)	ND	-25	ND	AB

a) D 5133: gelation index (G_i^{SB}) and gelation index temperature (T^{Gi}); b) D 4684: yield stress (σ_o^M), viscosity (η^M), and TP-1 temperature (T^M); c) engine borderline pumping temperature (T^{BP}); d) primary engine failure mode: air-binding (AB) or flow-limited (FL); e) not determined (ND)

We have undertaken an investigation into low-temperature rheological properties of lubricating mineral oils in order to understand mechanisms governing how pour-point depressants interact with wax crystallization processes and how this ultimately determines the rheology. As part of this effort, Webber [*8,9*] has reported recently on stress and temperature history effects on the low-temperature rheology of unadditized mineral oils; the key findings of this discussion are pertinent to addressing issues raised here about the gelation index parameter and its relation to low-temperature rheology.

Webber [*8*] found that the evolution of oil viscosity with decreasing temperature (i.e., a constant shear rate measurement analogous to the SBT) was characterized by a transient region where viscosity increased strongly with decreasing temperature, i.e., shows a large activation energy. The onset temperature (T_c) to this region was the onset temperature for formation of microscopically visible wax crystals. For a given oil, and therefore a given *n*-paraffin composition and thermodynamic description of the oil, the activation energy for viscosity as a function of temperature was observed to increase with increasing cooling rate. This corresponded to a decrease in the average wax crystal length, a strong increase in the apparent steady-state viscosity-stress behavior of the suspension, and a weak increase in yield stress. The effects of temperature history, specifically cooling rate during the onset of crystal nucleation and growth, was therefore the primary determinant of $T << T_c$ rheology for a particular oil. This behavior suggests that the wax crystal dispersion that results at low temperatures, $T << T_c$, is formed in two distinct stages, a rapid rate dependent nucleation and growth stage, and a rate independent stage. The former stage corresponds to the relief of supersaturation built up in the oil prior to crystallite nucleation.

The characterization of the behavior described above and the fact that it was dependent on the particular composition of the oil, most importantly the *n*-paraffin components, was also confounded by the effects of stress history. At $T < T_c$, the rheology was observed to be stress history dependent. Stress history appears to principally affect macroscopic structures that result when growing wax crystals interact and not the morphology of individual crystallites as determined from steady-state rheology [*8,9*].

The objective of this paper is to show how these findings apply to understanding the physical and rheological underpinnings of the gelation index as defined in D 5133. First we show how gelation index is associated with the onset region for wax crystallization rather than the actual onset of "gelation" within the oil. Second, we illustrate how stress history affects the instantaneous rheology and therefore the value of the gelation index. Then, by showing how rheology is affected by altering the *n*-paraffin composition of mineral oils in a controlled manner, we illustrate further the relationship between low-temperature rheology and nucleation processes within the oil, and most importantly, the non-unique aspects of the gelation index parameter. We conclude our discussion by showing that these results apply to formulated oils as well.

Experimental

Oil rheology was measured with a Carri-Med controlled stress rheometer over a temperature range from 20 to –35 °C, at constant cooling rates ranging from 0.5 to 60 °C/h. All measurements were made in a concentric cylinder geometry with temperature controlled at the cup surface. A comprehensive description of the rheological apparatus and specific procedures was given elsewhere [*8*]. In performing the rheological experiments we followed many of the recommendations outlined by Wardhaugh and Boger [*10,11*].

Viscosity (η) as a function of temperature (T) was measured by continuously shearing the oil at a constant shear rate ($\dot{\gamma}$) throughout the constant rate cooling cycle. The shear rate was typically 2 s^{-1}. η(T) curves measured at constant shear rates of 5 and 20 s^{-1} are reported here also.

Yield stress (σ_o) at a particular T was measured after cooling oil samples at a constant rate with a quiescent stress history, that is the oil was not strained ($\gamma = 0$) throughout the cooling cycle. σ_o was then determined from either a flow curve or oscillatory torque sweep experiment. Flow curves were measured by ramping stress logarithmically from 0.1 to 1200 Pa over a 10 min interval, and σ_o was defined as the limiting value for stress at $\dot{\gamma} = 0.01$ s^{-1}, i.e., the stress required to reach $\gamma = 0.01$ on a time scale of order 1 s. Torque sweep experiments consisted of measuring elastic (G') and loss (G") shear moduli at constant angular frequency ($\omega = 2\pi$ rad/s) as a function of applied stress. σ_o was defined as the onset stress for the rapid decrease in elastic modulus that indicates fracture of the macroscopic wax crystal structure [*9,12*].

At $T < T_c$, the rheology is stress history dependent. In previous work [*8*], we determined that at $T << T_c$, the wax crystal dispersion can be brought to apparent steady-state flow behavior by applying an extended stress history to the oil sample. Steady-state flow curves were then measured by performing a series of creep (constant stress as function of time) experiments over a series of increasing applied stresses (σ). Steady-state flow behavior was not dependent on the duration of the creep step. It was also independent of the concentric cylinder annulus gap (500 and 1000 μm) which indicates that the strong shear thinning behavior observed was not due to wall slip.

The mineral oils studied were two widely available, commercial API group I and II 100 neutral base oils; they are referred to as *I* and *II*, respectively. The effects of pour-point depressant concentration were studied by adding a poly(alkylmethacrylate) pour-point depressant to *II*-a (a different lot of oil *II*) that contained a GF-2 quality additive package but no viscosity modifier. All experiments were conducted with oil samples that were pretreated by heating to 90 °C for 1 to 2 h in a closed glass container (similar to that in D 4684 and D 5133). The heating minimizes effects of previous low temperature histories, i.e., from prior unspecified cooling or storage, by dissolving residual wax crystals. When subjected to the pretreatment, the rheology presented in this paper is reproducible.

A fraction of the wax forming components was separated from oil *II* by a solvent dewaxing procedure [*13*], specifically a procedure similar to UOP Method 46-85 (Paraffin Wax Content of Petroleum Oils and Asphalts, UOP Inc., Des Plaines, IL, 1985). The molecular weight distribution of the separated fraction of the wax forming components was obtained from gas chromatographic analysis; details of the separation procedure and chromatographic techniques are described elsewhere [*8*]. Under the conditions of the separation, 0.7 $\%_w$ of wax was separated from oil *II*. The amount of wax present in the oils at –35 °C was estimated using an NMR technique based on methods described by Pedersen et al. [*14*]; details of the procedures are described by Webber [*8*]. The wax content of the oils at –35 °C after 10 °C/h cooling was estimated as 15.7 and 9.5 $\%_w$ for oils *I* and *II*, respectively.

Results and Discussion

Effects of Wax Crystallization on Rheology

Viscosity (η) as a function of temperature (T) for oils *I* and *II* is shown in Figure 1 in a standard Andrade plot format where the activation energy (E) is proportional to the slope of the curve. For convenience, the upper x-axis shows the corresponding temperatures in Celsius. Viscosity was measured by continuously shearing the oil at a constant shear rate ($\dot{\gamma}$ = 2 s^{-1}) while the oils were cooled at a constant rate (b = 10 °C/h). This measurement is analogous (i.e., different constant shear rate and cooling rate) to the Scanning Brookfield test (SBT) described in D 5133, albeit, with significantly greater viscosity measurement range. The hatched viscosity range on the figure indicates the maximum reliable viscosity measurement range for the Brookfield viscometer specified for D 5133.

The mineral oils exhibited Newtonian rheology at room temperature. As temperature was decreased, an abrupt transition occurred to a region in which viscosity increased strongly with temperature, i.e., a high apparent activation energy ($E_{(i)}$). The onset temperature to this region is marked in the figure with an arrow labeled T_c. With further decrease in temperature, the activation energy relaxed to an approximately cooling rate independent value for the remainder of the temperature range. T_c was identified with the onset of microscopically visible crystallization of waxes within the oil [*8*]; this is illustrated in the inset to Figure 1 which shows the correspondence between

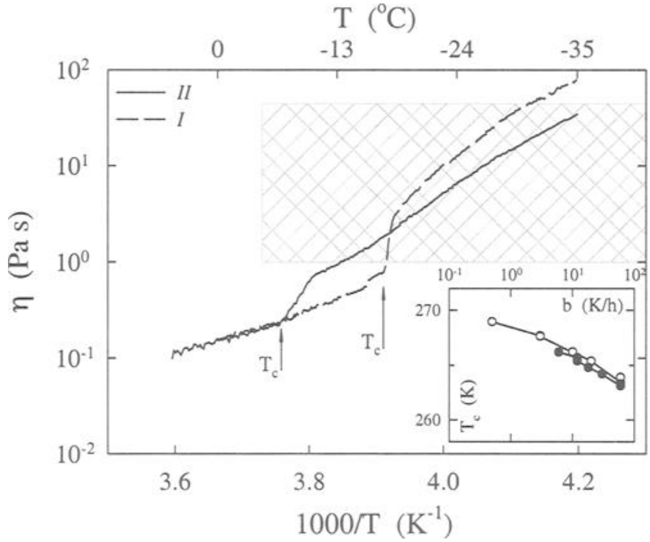


Figure 1 - *Viscosity (η) as a function of temperature (T) for oils I and II ($\dot{\gamma}$ = 2 s^{-1}, b = 10 °C /h). The inset shows onset temperature (T_c) as a function of cooling rate (b) for oil II from microscopy (filled symbols) and rheology (open symbols). The hatching indicates the viscosity measurement range of the Scanning Brookfield rheometer in D 5133.*

T_c measured by rheology and microscopy at different cooling rates for oil *II*. Similar behavior was observed with oil *I*.

The inset to Figure 1 shows that T_c is depressed by increasing the cooling rate. This behavior is expected for crystallization from solution [*15,16*] and may be indicative of the nucleation mechanism for wax crystallite formation. In previous work [*8*], we presented an extensive investigation into this mechanism and its associated rheology; it included η(T) measurements such as those shown in Figure 1, over an extensive 0.5 to 60 °C/h range of cooling rates. Over this range of cooling rates, η(T) behavior was similar to that illustrated in Figure 1, however, with increasing cooling rate T_c was depressed and the apparent viscosity-temperature activation energy ($E_{(i)}$) increased. This and other data presented in that work [*8*], including wax crystal size measurements as a function of cooling rate, led us to two important conclusions. First, T_c is the onset temperature for the start of crystal nucleation and growth. Second, the strong viscosity-temperature activation energy region is associated with the relief of supersaturation that builds upon cooling below the saturation temperature for the *n*-paraffinic components of the oil. The development of supersaturation and its relief through nucleation and crystal growth depend on both the molecular weight distribution and concentration of *n*-paraffinic components within oil as well as the composition of the oil [*15-17*]. Therefore, this process determines the activation energy in the vicinity of T_c.

The D 5133 definition for gelation index (G_i^{SB}) can be applied to the $\eta(T)$ curves for oils *I* and *II* shown in Figure 1. With respect to eq (1), G_i can be calculated from the maximum activation energy ($E_{(i)}$) in the Andrade plot

$$G_i = \left(1 + \frac{A_{(i)}T}{E_{(i)}}\right)^{-1} \quad (2)$$

where $A_{(i)}$ is the intercept from the linear fit used to determine $E_{(i)}$. The maximum $E_{(i)}$ occurs in the cooling rate dependent nucleation regime. The maximum value of G_i occurs at T_c, the onset temperature for crystal nucleation and growth. Under the conditions in Figure 1, T_c was -17.3 and -6.8 °C and G_i was calculated as 52.7 and 17.5 for oils *I* and *II*, respectively. With respect to the limitations of the Scanning Brookfield instrument, the nucleation region for oil *II* could not be observed while that for the oil *I* was only partially visible; accordingly, G_i values of 126.8 and 7.3 were reported as measured by D 5133 for *I* and *II*, respectively. Since these are both unadditized oils, they exhibit significant yield stresses at – 35 °C, 1150 and 556 Pa for oils *I* and *II*, respectively.

Optical micrographs showed that as temperature was decreased below T_c, the size and number density of crystallites increased. In the vicinity of T_c, crystals were sparsely located. At temperatures well below T_c, crystals appeared to grow to large enough sizes such that they physically interacted with neighboring crystals. Development of solid-like rheology as implied by the term gelation requires that crystals interact and form structures on length scales relevant to macroscopic rheological measurements. For example, structures formed from collections of wax crystals must percolate (by forming a wax crystalline lattice) through length scales on the order of the gap dimension of the rheometer in order to support a stress that is consistent with a yield stress. Micrographs published earlier [*8*] of oil *II* cooled at a rate of 6 °C/h suggest that the transition to percolation occurs between –18 and –25 °C, significantly lower than T_c.

The onset for macroscopic structure formation and gelation was studied explicitly by measuring yield stress (σ_o) as a function of temperature at a constant cooling rate (b = 10 °C/h). Oils *I* and *II* were cooled quiescently to the measurement temperature and yield stress was determined from oscillatory torque sweep experiments. Figure 2 shows σ_o as a function of T where T_c values from Figure 1 are indicated by arrows on the abscissa. Yield stress increased strongly with decreasing temperature. For both oils, significant yield stresses (i.e., significant with respect to the order 1 s experiment time scale is approximately 10 Pa) were not observed until the temperature was well below T_c. For example, at –15 °C, oil *II* exhibited loss dominant (viscous) behavior suggesting that wax crystals have not grown yet to the size and number required to form the wax crystal lattice structure necessary for dominant solid-like elastic behavior. The hatched region in the figure indicates failing yield stresses for the MRV TP-1 test (D 4684); it shows that MRV failing yield stresses would not be recorded until T < -20 °C for both oils. The rheology is therefore consistent with the conclusions reached from the photomicrographs, i.e., that the required macroscopic wax crystal structures develop as crystal number density and size increase to the point where crystals interact and eventually percolate across macroscopic dimensions. This state does not occur until temperatures well below the onset of crystal nucleation and thus serves to disconnect G_i, i.e., the strong viscosity increase with temperature, from a technical definition of gelation.

Effects of Stress History

The yield stress measurements shown in Figure 2 were sensitive to the stress or strain (γ) history imposed on the sample. Strain histories as small as $\gamma = 0.01$ were greater than the characteristic yield strain for wax crystal structures formed in oils *I* and *II* and thus affected the rheological properties of developing wax crystal structures [*9*]. Webber also showed that the stresses imposed during the cooling cycle of the $\eta(T)$ measurements such as that of Figure 1, i.e., significant permanent strain deformations due to the continuous shear rate, precluded measurement of any significant yield stress and thus any solid-like structure development within oils *I* and *II*. In addition, structure and yield stresses were not recovered even after significant wait times at the final low temperature, consistent with estimated low Brownian diffusivities for crystals within the oil at –35 °C.

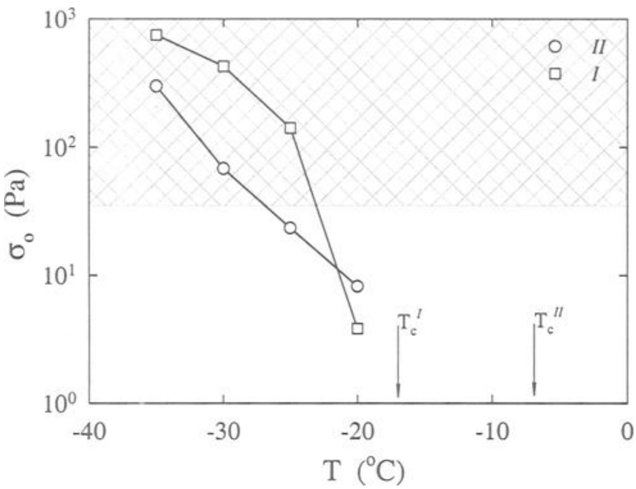


Figure 2 - *Yield stress (σ_o) as a function of T for oils I and II (quiescent cooling ($\gamma = 0$), b = 10 °C/h). The hatching indicates failing yield stresses for MRV TP-1 in D 4684.*

Stress history through the constant applied shear rate also had subtle effects and yet profound implications for interpreting the $T < T_c$ rheology in $\eta(T)$ constant shear rate experiments. Figure 3 shows $\eta(T)$ measured at various constant shear rates for oil *II* samples cooled at constant rate (b = 10 °C/h). At $T > T_c$, the oil was Newtonian. T_c was independent of shear rate. However, viscosity at $T < T_c$ decreased with increasing shear rate, showing apparent shear thinning behavior. Activation energy ($E_{(i)}$) for $\eta(T)$ in the nucleation regime, and therefore G_i, decreased strongly with increasing shear rate. Table 2 lists values for G_i determined using eq (2).

At $T < T_c$ and constant cooling rate, the steady-state flow behavior of the wax crystal dispersion was a function of the volume fraction of crystallized wax, crystallite shape morphology, and the size distribution of crystallites [*8*], properties which are a

Table 2 - *Effect of stress history (i.e., at various constant shear rates,* $\dot{\gamma}$*) on gelation index* (G_i) *for oil II (b = 10 °C/h, see Figure 3).*

$\dot{\gamma}$ (s^{-1})	G_i
2	17.5 ±1.2
5	9.6
20	8.5

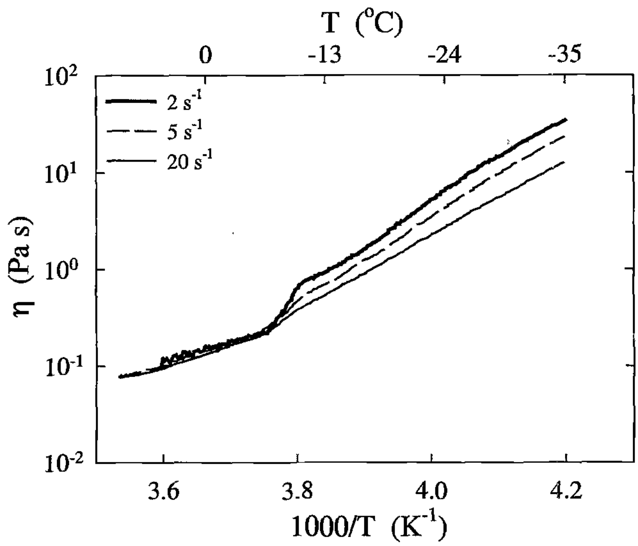


Figure 3 - *η as a function of T for oil II at various constant shear rates (b = 10 °C/h).*

function of temperature and cooling rate. Flow properties of the dispersion were also sensitive to the state of crystallite agglomeration, i.e., an effective volume fraction, as well as the degree of flow induced alignment of crystallites. These latter aspects likely contribute to produce the observed stress history effects; it is the latter state that is modified by the imposed stress as the dispersion transitions from stress history dependent to steady-state flow behavior. The principle effect of stress history at $T < T_c$ was to affect how flow rheology evolves with applied stress over time (i.e., towards the apparent steady-state behavior). The stress history affected the macrostructure of wax crystallites in the dispersion, but not the microstructure or morphology of individual wax crystallites. The steady-state flow curves were, therefore, independent of the stress history; for example, the three $\eta(T)$ measurements shown in Figure 3 produced the same steady-state flow curves [*8*]. The steady-state flow behavior did, however, depend strongly on temperature history (cooling rate) and this has been correlated with how temperature history affected the wax crystal size distribution; increasing cooling rate decreased average crystal size which strongly increased viscosity in the low shear rate limit and increased the non-Newtonian character of the flow curve [*8*]. Characterization of instantaneous flow behavior such as in $\eta(T)$ measurements in Figure 1 and with the SBT (and from that $E_{(i)}$ and G_i) is dependent on the prior stress history. Therefore, despite the constant shear rate, the stress history is different for each oil since the stress history evolves concurrently with the dispersion macrostructure according to the wax crystal nucleation and growth processes, its evolution is coupled to the instantaneous oil rheology. The G_i parameter is thus a non-unique characterization of this process as shown in Figure 3 and Table 2; it is highly dependent upon one's choice of test conditions.

The broader implication of this result is that it is inappropriate to compare G_i values for different composition oils. This can be understood by considering how compositional differences, especially with respect to the molecular weight distribution of *n*-paraffinic components, affect the development of supersaturation, wax crystal nucleation and crystal growth processes in oils. Since each oil undoubtedly has a unique *n*-paraffin molecular weight distribution and composition, it has a unique distribution of saturation temperatures (T_{sat}) for the *n*-paraffinic components that will compose the wax [*17*]. The build up in concentration of supersaturated *n*-paraffinic components within the oil as it is cooled to temperatures below that of the distribution of saturation temperatures is dependent on the shape of the distribution; this determines the crystal nucleation and growth processes for that oil. The nucleation and growth processes determine the instantaneous rheology and the manner in which the oil responds to the stress history of the constant shear rate $\eta(T)$ measurement, and therefore, where the rheology is with respect to its steady-state rheology at $T < T_c$. A unique value of G_i, i.e., one that is characteristic of just the nucleation and growth process and not some arbitrary state of crystal agglomeration and alignment, is not measurable with this type of constant shear rate experiment. In the following, we illustrate these points in a direct manner by manipulating the composition of wax forming components within an oil.

Effects of n-Paraffin Composition

From NMR measurements, it was estimated that oil *II* contains approximately 9.5 $\%_w$ wax at –35 °C. The composition of wax forming components in oil *II* was altered by extraction and addition. A fraction of wax forming components was extracted by a solvent dewaxing method. Approximately 0.7 $\%_w$ wax was removed along with a small fraction of entrained oil which is assumed to be representative of the bulk oil. 1 $\%_w$ of an oil-free paraffinic wax, Shellwax 100, was added to oil *II*. In both cases, dewaxed and added-wax, the resultant oil was preheated to 90 °C prior to rheological experimentation.

Figure 4 shows effects of dewaxing (sample: *II* - wax) and Figure 5 the effect of added-wax (sample: *II* + wax) on η(T) measured at constant shear rate ($\dot{\gamma}$ = 2 s^{-1}) and constant cooling rate (b = 10 °C/h). The molecular weight distribution of the wax removed from the oil is shown in the inset to Figure 4 and that for the wax added to oil in the inset to Figure 5. Results are summarized in Table 3. Removal of the wax caused T_c to decrease, while addition of wax caused T_c to increase. The changes to T_c are qualitatively consistent with expectations for how changes in the wax component composition affect thermodynamic and kinetic descriptions of this system [*15-17*]. Figures 4 and 5 also show that viscosity decreased with the removal of wax components and increased with the addition of wax components. A substantial increase in G_i (see Table 3) was observed upon removal of wax while a weak decrease in G_i was observed for the sample with added wax. According to Selby [*3-5*], Gi correlates with low temperature engine oil pumpability so that increasing G_i, increases the tendency to gelation in the oil, and therefore, the potential for air binding. While the changes in viscosity are at least apparently consistent with expectations regarding effects of addition and extraction of wax forming components, the changes in G_i are not consistent with either the viscosity measurements or expectations.

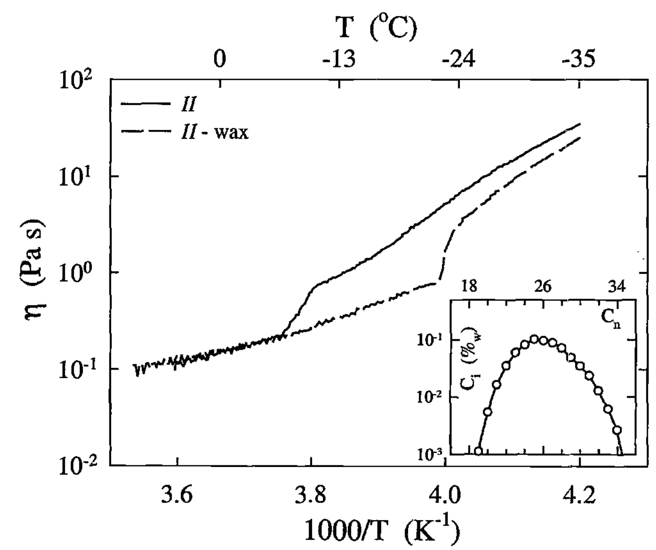


Figure 4 - η as a function of T for II, and dewaxed II (II - wax) ($\dot{\gamma}$ = 2 s^{-1}, b = 10 °C/h). The inset shows the molecular weight distribution of wax forming components removed from the oil (i^{th} component concentration (C_i) versus carbon number (C_n)).

Yield stress (σ_o) was measured using ramp stress measurements after quiescently cooling samples of *II* - wax and *II* + wax at 10 °C/h to –35 °C. Results for σ_o are listed in Table 3. σ_o decreased substantially upon removal of the wax, and weakly with addition of the wax. The corresponding steady-state flow behavior, i.e., viscosity (η) as a function of stress σ, at – 35 °C is shown in Figure 6. Removal of the wax substantially decreased viscosity, especially at low shear rates, while addition of the wax only weakly affected the flow curve. The T << T_c rheological measurements clearly do not support a positive relation, or for that matter any linear relation, between G_i and common

Table 3 - *Effect of removal and addition of wax fractions to oil II on gelation index (G_i) and yield stress (σ_o) (b = 10 °C /h, see Figures 4 and 5).*

sample	comments	T_c (°C)	G_i	σ_o (Pa) [b]
II	9.5 $\%_w$ wax at -35 °C [a]	-6.8	17.5 ±1.2	556 ±83
II – wax	0.7 $\%_w$ wax removed	-22.4	44.2	191
II + wax	1.0 $\%_w$ wax added	8.3	15.8	389

[a] determined from NMR measurements (b = 10 °C /h); [b] measured in separate experiments, see text for explanation

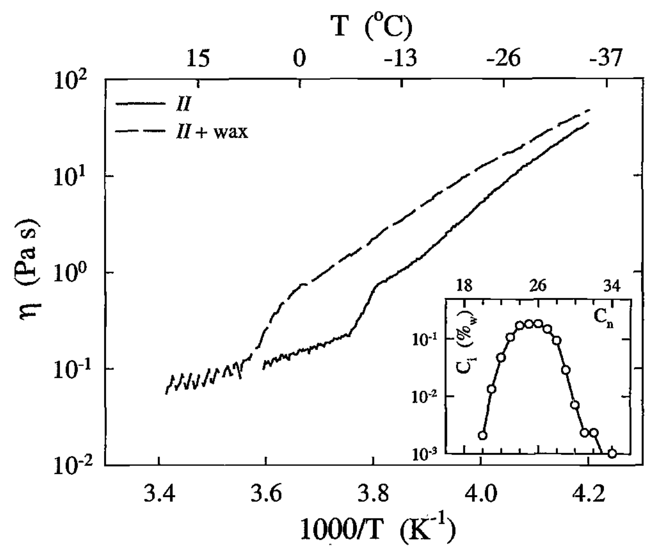


Figure 5 - *η as a function of T for II and II with added wax (II + wax) ($\dot{\gamma}$ = 2 s^{-1}, b = 10 °C/h). The inset shows the molecular weight distribution of wax forming components added to the oil.*

rheological measurements that might be associated with gelation tendency in the oil or pumpability in the engine, whether by instantaneous viscosity measurement (Figures 4 and 5), yield stress (Table 3), or steady-state flow behavior (Figure 6).

Effects of Additives and Pour-Point Depressant Concentration

Are these results and interpretations relevant to the behavior of formulated oils? To date, we have found that the behaviors described above are consistent with those observed in formulated oils. In fact, the behaviors observed with formulated oils also serve to reinforce conclusions about the non-unique nature of G_i and its dependence on the nucleation process.

Figure 7 shows the $\eta(T)$ response for the *II*-a oil containing a GF-2 quality additive package. $\eta(T)$ was measured at constant shear rate ($\dot{\gamma}$ = 2 s^{-1}) while the oil was cooled at b = 10 °C/h. *II*-a was from a different batch of oil than *II* used in studies reported above; the rheology shows that *II*-a probably contains a different *n*-paraffin molecular weight distribution than lot *II*. The 0.0 $\%_w$ $\eta(T)$ curve in Figure 7 is similar to that for oil *II* in Figure 1, however, T_c is shifted to a lower temperature, -10.6 °C. This shift was not caused by the presence of the additive package as evidence by similar experiments on oil *II*-a without the additive package. Table 4 shows that G_i for oil *II*-a was larger than that of oil *II*. Despite the increased G_i, both the yield stress (σ_o), shown in Table 4, and

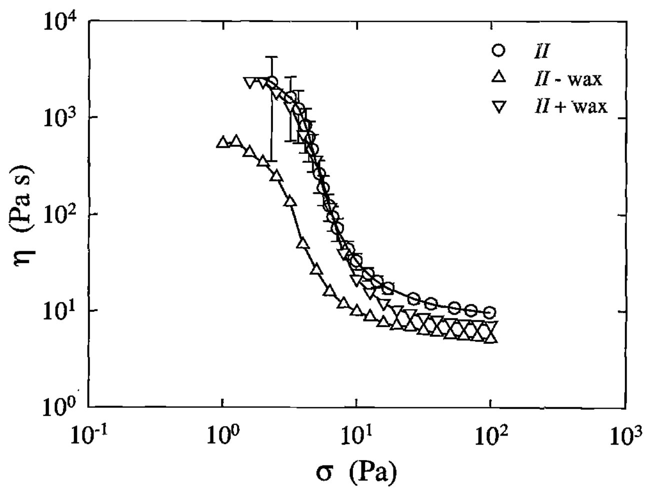


Figure 6 - *η as a function of stress (σ) at T = -35 °C for oil II samples shown in Figures 4 and 5 (apparent steady-state flow, b = 10 °C/h).*

steady-state flow behavior (not shown) were significantly lower for oil *II*-a as compared to oil *II*. This apparent disconnect between G_i and the observed rheology again highlights the non-unique nature of G_i discussed earlier.

The effects of pour-point depressant (PPD) concentration (C_{PPD}) on η(T) of the additized oil *II*-a are shown in Figure 7. In these studies, C_{PPD} ranged from amounts orders of magnitude lower than those found in commercial engine oils to more typical concentrations. At low C_{PPD}, it was possible to observe incremental effects of the PPD on the characteristic rheology described above. The effect of the PPD was to reduce the total increase in viscosity in the nucleation region at $T < T_c$ as illustrated with the 0.004 $\%_w$ curve. At lower C_{PPD} than illustrated here, the PPD had quantifiable affects on the rheology yet no apparent affect on $E_{(i)}$ and G_i. For the 0.004 $\%_w$ concentration, the

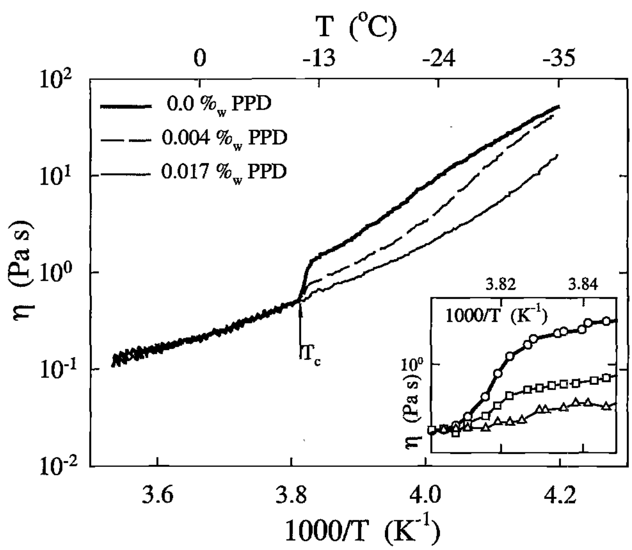


Figure 7 - *η as a function of T for oil II-a containing a GF-2 quality additive package and various concentrations of pour-point depressant (* $\dot{\gamma}$ *= 2* s^{-1}*, b = 10 °C/h). The inset shows an enlargement of the* T_c *region.*

activation energy in the nucleation region at $T < T_c$ was decreased. As can be seen from the inset figure which is an expanded view of the T_c and nucleation region, this may have been a result of the small number of measured viscosity values available in the transition region rather than due to a fundamental change in the nucleation process. At higher PPD concentrations, albeit still an order of magnitude lower than used in typical engine oil, T_c and the nucleation transition were no longer visible. The results shown here were not unique to the relatively high cooling rates (b = 10 °C/h) of these experiments; the same cooling rate effects reported in previous work [8] were observed in this system. The PPD appeared to affect the growth in viscosity after the onset of nucleation, it did not appear to affect the onset of the nucleation process and its apparent activation energy, hence the weak effects on G_i. This suggests a mechanism whereby this type of PPD affects low-temperature rheology by interacting with the growth process of already nucleated wax crystals. Micrographs of wax crystals support this interpretation since distinct changes in crystal morphology with increasing C_{PPD} were observed. At very low C_{PPD}, the effects on rheology appeared to be limited by the low concentration of PPD within the oil.

Table 4 shows σ_o values as a function of C_{PPD}. Yield stress decreased sharply with increasing PPD concentration and by 0.017 $\%_w$ PPD, yield behavior was no longer evident in quiescently cooled samples. Added PPD also affected the shear thinning nature of the steady-state flow curves shown in Figure 6. As PPD concentration was increased, the steady-state flow curve took on a more Newtonian character primarily through a reduction in the low shear rate, Newtonian plateau viscosity.

Table 4 - *Effect of pour-point depressant concentration (C_{PPD}) on gelation index (G_i) and yield stress (σ_o) for oil II-a containing a GF-2 quality additive package (b = 10 °C/h, see Figure 7).*

C_{PPD} ($\%_w$)	G_i	σ_o (Pa) [a]
0.0	31.6	327
0.004	17.5	90
0.017	4.0	0.7 [b]

[a] measured in separate experiments, see text for explanation (b = 10 °C/h); [b] no evidence of a yield stress

Conclusions

The objective of this paper was to discuss the physical and rheological behaviors of wax crystallization within oils, specifically with respect to its relation to the gelation index parameter. Through the use of controlled stress rheometric techniques we have significantly extended the constant shear rate viscosity-temperature measurement range beyond that of the Scanning Brookfield instrument. Upon cooling to the wax crystallization onset temperature (T_c), mineral oils containing *n*-paraffinic components showed a high activation energy region. The high activation energy was associated with the relief of supersaturation accrued within the oil due to its cooling to temperatures below that of saturation temperatures for *n*-paraffinic components of the oil. Gelation index defined according to D 5133 was determined by the activation energy in the nucleation region, and the gelation index temperature corresponded to T_c.

At $T < T_c$, rheology was stress history dependent. Stress history appeared to principally affect macroscopic structures that result when growing wax crystals interact and not the morphology of individual crystallites. The instantaneous flow behavior, e.g., as in $\eta(T)$ measurements of the Scanning Brookfield test, therefore was dependent on the stress history. By definition, stress history is not controlled in an instantaneous constant shear rate measurement. Gelation index is thus necessarily a non-unique characterization of the wax crystallization process.

The implication is that it is inappropriate to compare gelation index values among oils of different composition since the nucleation process and its subsequent sensitivity to stress histories are uniquely different for each oil. This was illustrated by manipulating the composition of wax forming components within an oil and determining how that affected the activation energy in the nucleation regime as well as other low-temperature rheological properties such as yield stress. The *n*-paraffin composition of an oil was changed both by dewaxing as well as by addition of wax components. Dewaxing depressed T_c while wax addition increased T_c, consistent with expectations based on thermodynamic and nucleation theories. Gelation index increased upon dewaxing and decreased upon addition of wax. According to explanations of the gelation index characterization provided by Selby [5], these changes in gelation index indicate that removal of wax should increase the gelling tendency of the oil. This was not consistent with the observed low-temperature rheology. For example, yield stress decreased substantially with dewaxing in contrast to the observed increase in gelation index.

The final section of this paper showed that the same rheological interpretations also applied to the low-temperature rheological behavior of formulated oils and the subsequent effects of pour-point depressant polymers on their behavior. The formulated oils showed a high activation energy region similar to that of simple mineral oils. The effect of pour-point depressant was to reduce the extent of viscosity change that occurred in the nucleation and growth region; the activation energy for viscosity as a function of temperature was only weakly affected where the accuracy of this characterization was limited by narrowing of the temperature range. Over this concentration range, the addition of pour-point depressant significantly decreased the yield stress and the limiting low shear rate viscosity.

Acknowledgments

The authors thank K. W. Raybuck for the optical microscopy, B. L. Soukup for image analysis of the micrographs, D. A. Ebner for developing the wax separation technique, and K. F. Wollenberg for the NMR measurements. The authors also thank T. Selby for supplying the PRO oils for which additional MRV and SBT results were reported.

References

[1] Henderson, K. O., Manning, R. E., May, C. J., and Rhodes, R. B., "New Mini-Rotary Viscometer Temperature Profiles that Predict Engine Oil Pumpability," SAE Technical Paper No. 850443, 1985.

[2] Shaub, H., "A History of ASTM Accomplishments in Low Temperature Engine Oil Rheology: 1966-1991," *Low Temperature Lubricant Rheology Measurement and Relevance to Engine Operation," ASTM STP 143*, R. B. Rhodes, Ed., American Society for Testing and Materials, West Conshohocken, PA, 1992, pp. 1-19.

[3] Selby, T. W., "Further Considerations of Low-Temperature, Low Shear-Rate Rheology Related to Engine Oil Pumpability – Information from the Scanning Brookfield Technique," SAE Technical Paper No. 852115, 1985.

[4] Selby, T. W., "The Use of the Scanning Brookfield Technique to Study the Critical Degree of Gelation of Lubricants at Low Temperatures," SAE Technical Paper No. 910746, 1991.

[5] Selby, T. W., "The Scanning Brookfield Technique of Low-Temperature, Low-Shear Rheology – Its Inception, Development, and Applications," *Low Temperature Lubricant Rheology Measurement and Relevance to Engine Operation*," *ASTM STP 143*, R. B. Rhodes, Ed., American Society for Testing and Materials, West Conshohocken, PA, 1992, pp. 33-64.

[6] Kinker, B. G., Souchik, J. M., and Neveu, C. D., "Evaluation of the Low Temperature Performance of Engine Lubricants Using the Scanning Brookfield Viscometer," *Proceedings of 11th Intern. Colloquium on Industrial and Automotive Lubrication*, W. J. Bartz, Ed., Vol. II, Technische Akademie Esslingen, 1998, pp. 1419-1426.

[7] Koenitzer, B. A., Lai, P. K. S., and May, C. J., "A Study of Gel Index and MRV Effects on SAE 5W-30 Pumpability," SAE Technical Paper No. 982641, 1998.

[8] Webber, R. M., "Low Temperature Rheology of Lubricating Mineral Oils: Effects of Cooling Rate and Wax Crystallization on Flow Properties of Base Oils," *Journal Rheology*, 1999, 43, 911-931.

[9] Webber, R. M., "Rheological Characterization of Wax Crystal Structuring in Mineral Oils: Effects of Stress and Temperature History," *International Conference on Petroleum Phase Behavior and Fouling*, AIChE Topical Conference Preprints, Houston, TX, 1999, pp. 138-144.

[10] Wardhaugh, L. T. and Boger, D. V., "Measurement of the Unique Flow Properties of Waxy Crude Oils," *Chemical Engineering Research Design*, 1987, 65, 74-83.

[11] Wardhaugh, L. T. and Boger, D. V., "Flow Characteristics of Waxy Crude Oils: Application to Pipeline Design," *AIChE Journal*, 1991, 37, 871-885.

[12] Chang, C., Boger, D. V., Nguyen, Q. D., "The Yielding of Waxy Crude Oils," *Industrial Engineering Chemistry Research*, 1998, 37, 1551-559.

[13] Passut, C. A., Barton, P., Klaus, E. E., and Tewksbury, E. J., "Low Temperature Ketone Dewaxing of Mineral Oils by Direct Cooling," *Industrial Engineering Chemistry, Process Design Development*, 1977, 16, 120-124.

[14] Pedersen, W. B., Hansen, A. B., Larsen, E., Nielsen, A. B., and Ronningsen, H. P.

"Wax Precipitation from North Sea Crude Oils. 2. Solid-Phase Content as Function of Temperature Determined by Pulsed NMR," *Energy & Fuels*, 1991, 5, 908-913.

[15] Walton, A. G., "Nucleation in Liquids and Solutions" *Nucleation*, A. C. Zettlemoyer, Ed., Marcel Dekker, New York, NY, 1969, pp. 379-404.

[16] Mullin, J. W., *Crystallization*, 3rd Edition, Butterworth Hinemann, Oxford, 1993.

[17] Lira-Galeana, C., A. Firoozabadi, J. M. Prausnitz, "Thermodynamics of Wax Precipitation in Petroleum Mixtures," *AIChE Journal*, 1996, 42, 239-248.

Christopher J. May,[1] Bernie A. Koenitzer,[2] and Patrick K. S. Lai[3]

SAE 5W-30 Pumpability Studies in Modern 4- and 8-Cylinder Engines: Gelation Index and MRV Effects

---

Reference: May, C. J., Koenitzer, B. A., and Lai, P. K. S., "**SAE 5W-30 Pumpability Studies in Modern 4- and 8-Cylinder Engines: Gelation Index and MRV Effects,**" *Oil Flow Studies at Low Temperatures in Modern Engines, ASTM STP 1388,* H. Shaub, Ed., American Society for Testing and Materials, West Conshohocken, PA, 2000.

Abstract: Four SAE 5W-30 formulations with a range of MRV and Gelation Index properties were tested in motored 4- and 8- cylinder engines at ambient temperatures between -35°C and -38°C (below anticipated minimum start temperatures (MSTs)). A slow-cooling profile was used to enhance gelation effects in the test engines, which were motored at normal fast idle speeds. Oil pressurization after the pump was relatively rapid in all cases and did not show a large dependence on oil type or temperature. However, pressurization times at the main gallery showed a correlation to interpolated D 4684 MRV viscosities of the test oils. No correlation was observed between pumpability characteristics and D 5133 gelation index. While the two 2.0L I-4 engines gave comparable pressurization characteristics, the two 4.6L V-8s were quite different from each other. Pumpability differences between the V-8 engines were due to the presence of a plate-type oil cooler in one engine, which reduced oil pressure by 200 KPa and lead to significantly longer pressurization times. At the lowest test temperatures, the 2.0L designs showed 'pseudo air-binding' behaviour with all the test oils, in which gallery pressure dropped near zero after an initial pressure spike; pressure before the filter, however, continued to be registered. Low temperature rheological analysis of some of the used test oils was conducted to understand changes occurring after the relatively brief engine operation. In some cases oils with higher gelation indices showed significant decreases after engine operation, while MRV values were relatively unaffected.

Keywords: pumpability, light duty engines, MRV, gelation index, pumping viscosity, air-binding failure, flow-limited failure.

---

[1] Senior Research Associate, Imperial Oil Research Dept., P.O. Box 3022, Sarnia, Ont., Canada N7T 8C8.

[2] Senior Research Technologist, Imperial Oil Research Dept., P.O. Box 3022, Sarnia, Ont., Canada N7T 8C8.

[3] Section Head, Automotive Test Section, Imperial Oil Research Dept., P.O. Box 3022, Sarnia, Ont., Canada N7T 8C8.

Introduction

Low temperature operability of light- and heavy-duty equipment has been a concern for original equipment manufacturers (OEMs) and oil formulators alike for many years. In the low shear, low temperature regime, two procedures are commonly employed to assess lubricant pumpability characteristics of engine oils: Determination of Yield Stress and Apparent Viscosity of Engine Oils at Low Temperature (D 4684) [*1*], and Low Temperature, Low Shear Rate, Viscosity/Temperature Dependence of Lubricating Oils Using a Temperature-Scanning Technique (D 5133)[*2-5*]. Limits for the former test have been tightened to reflect the generally greater ease of cold-start characteristics of modern engines, and are contained in the SAE Engine Oil Viscosity Classification standard J300; the test assesses both flow-limited (viscosity) and air-binding (yield stress) tendencies of the lubricant (Figure 1). Limits for D 5133 gelation index have been established within the ILSAC GF-2 specification at 12 max [*6*], although at least one OEM has an 8.5 maximum limit for factory and service fill engine oils. Gelation index is considered to relate to air-binding tendencies in engines [*4,5*].

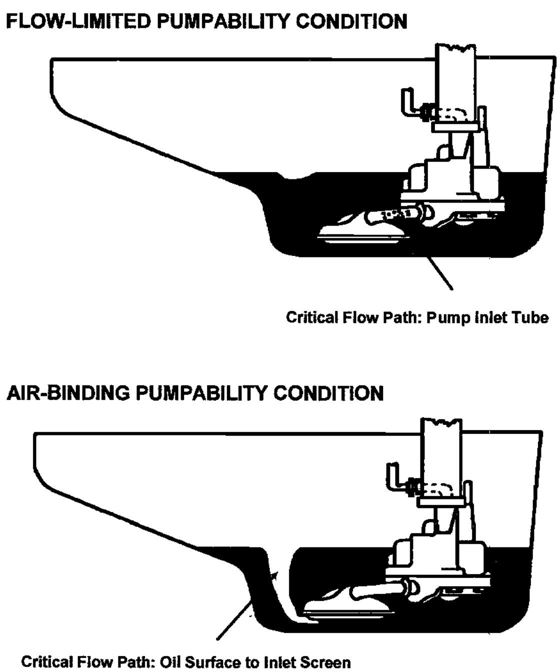


Figure 1 - *Schematic of Flow-Limited and Air-Binding Pumpability Failure Mechanisms*

Although it has been shown to be irrelevant to engine pumpability, the D97 pour point test continues to be used by the industry as a measure of dispensability at low temperatures, and most oil producers have internal specifications for this parameter. Balancing all three parameters: MRV, gelation index and pour point, can be a substantial challenge for oil formulators since the tests measure performance under very different combinations of cooling rate, shear rate and shear stress (Table 1).

Table 1: *Comparison of Cooling Rate/Range, Shear Rate and Shear Stress for Common Engine Oil Low Temperature Tests*

Test	Key Cooling Rate, °C/hr	Cooling Range (Slow Cool), °C	Shear Rate, sec^{-1}	Shear Stress, Pa
D4684 (TP-1 MRV)	0.5 to 0.33	+80 to -40 (-5 to -20)	0.4 to 200	525*
D5133 (Scanning Brookfield Gel Index)	1	+90 to -40 (-5 to -40)	0.17	1.7 - 8.5
D97 (Pour Point)	~25 - 30	+40 to <-54	Very low	Very low

* Viscosity Measurement only.

An example of the problem in balancing these low temperature properties is illustrated in Figure 2[4], where data for MRV viscosity and gel index values are compared in a series of 5W-30 and 10W-30 blends with SJ/GF-2 performance packages.

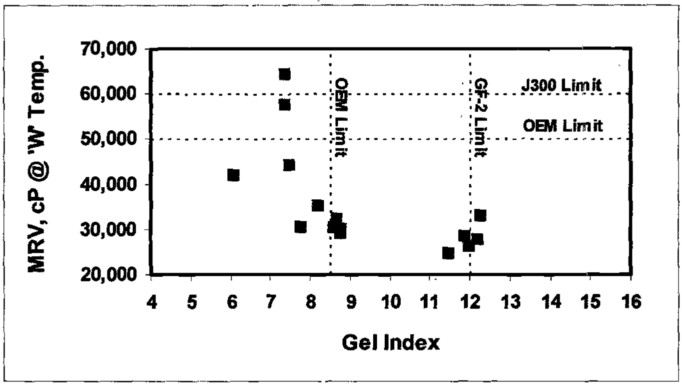


Figure 2 - *Gelation Index vs MRV Viscosity, Series of 5W-30 and 10W-30 Oils*

It is apparent that MRV viscosity and gelation index can vary independently of each other, and that an oil may fail one specification while passing the other. The same is true if a measure of air-binding tendencies as determined by the two test methods are compared (ie. gelation index versus yield stress - Figure 3). Similar observations have been reported in the open literature [*7,10*].

[4] Throughout this paper, viscosity data are referenced in units of cSt (kinematic), and cP (dynamic); conversion to SI Units is: 1 cSt = 1 mm^2/s, 1 cP = 1 mPa•s.

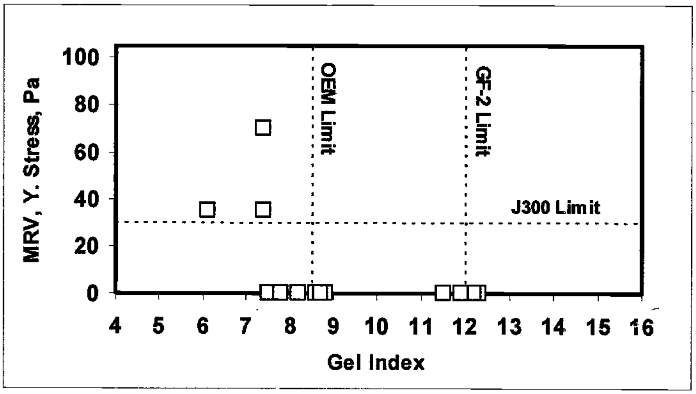


Figure 3 - *Gel Index vs MRV Yield Stress: 5W/10W-30s (Y. Stress<35 Pa shown as 0)*

Engine Pumpability Studies

Test Oils

In order to more fully understand the real world implications of differences between MRV and gel index in formulated oils, full scale engine pumpability testing was conducted on four SAE 5W-30 engine oils, whose properties are outlined in Table 2 [*10*].

Table 2: *SAE 5W-30 Pumpability Test Oils*

Inspections	Oil 'A'	Oil 'B'	Oil 'C'	Oil 'D'
KV100, cSt	10.99	11.01	10.92	10.27
CCS, cP @ -25°C	2,630	2,680	2,790	2,770
D4684 MRV, cP @ -35°C (yield stress, Pa)	28,000 (<35)	28,400 (<35)	26,800 (<35)	31,800 (<35)
D4684 MRV, cP @ -40°C (yield stress, Pa)	126,100 (35)	130,550 (<35)	97,350 (<35)	164,350 (35)
D5133 Gel Index	15.7	12.4	9.2	6.0
Gel Index Temperature, °C	-23	-23	-24	-35

Oils 'A' and 'D' readily met the MRV requirements for 5W oils (60,000 cP max. @ -35°C, <35 Pa) as well as the more severe OEM limit of 50,000 cP max. The gel index values for these oils, however, would suggest potential gelation issues: both were above the GF-2 maximum with Oil 'A's value close to the originally proposed 16 max. limit (*5*), and 'B' closer to the GF-2 specification. Oil 'C' was slightly better in MRV properties than the first 2 oils, and also met the GF-2 gel index specification; this oil would still fail the OEM requirement of 8.5 max GI. The fourth oil, 'D', had a gel index value meeting both the GF-2 limit and the more severe OEM requirement. While its MRV performance was acceptable by either OEM or J300 standards, it was directionally poorer than the other three oils.

Test Engines

For this pumpability work, full-scale vehicles were utilized, equipped with the appropriate engines and transmissions:

- Type I: 1997 Model Year Vehicle equipped with 2.0L I-4 engine and 5-speed manual transmission. 2 identical cars were used.
- Type II: 1997 Model Year Vehicle equipped with 4.6L V-8 engine and 5-speed manual transmission. 2 cars used for the test, one equipped with oil-cooler (Type IIB).

Vehicles were selected on the basis of similar types having been used under the auspices of the ASTM D.02.07 Low Temperature Engine Performance (LTEP) program [*9*]. This allowed an up-front assessment of pumpability hardware characteristics and estimates for appropriate pumpability testing temperatures, given the expected minimum starting temperatures. These properties are summarized in Table 3[5].

Table 3: *Hardware and Cold Starting Characteristics of Test Engines*

	Vehicle Type I	Vehicle Type II
Engine	2.0L I-4	4.6L V-8
Fast Idle Speed, cold start*, rpm	1500	1100
Oil Height, cm above pick-up	4.6	10.7
Pickup tube Length/Diameter^4	1.6	1.7
MST, °C, SAE 5W-30 oil*	-32.7	-31.1

* Based on Imperial Oil data provided to ASTM LTEP

Test Facility

- All Weather Chassis Dynamometer (AWCD) with separate presoak room. Computer controlled, two stage cooling compressor system. Capable of controlled cooling rates as slow as 0.1°C/hr. Motoring/Absorbing Dynamometer: dual 112 kW (150 H.P.) 1.2 m roller diameter.
- Engines motored via drive wheels with dynamometer to normal fast idle speed within 10-20 seconds of start-up; maximum 400 seconds running time.

Instrumentation (see Schematic, Figure 4)

- 3 thermocouples in oil pan (3 different depths; middle T/C next to pick-up screen).
- Vacuum transducer located in pick-up tube. Pressure transducer before oil filter ('To Filter') and in normal sensor location ('Gallery'). Note that Vehicle 4 was equipped with a 12 plate, single pass oil cooler, hence the 'to filter' pressure sensor was located after the oil cooler, but before the filter (see Figure 5).

[5] Information taken from Imperial Oil test results provided to ASTM D.02.07 LTEP program [*9*].

- Engine speed obtained from engine monitoring system.
- Data acquisition: 2 times per second during pumpability; temperature acquisition 5 times per hour during cooldown.

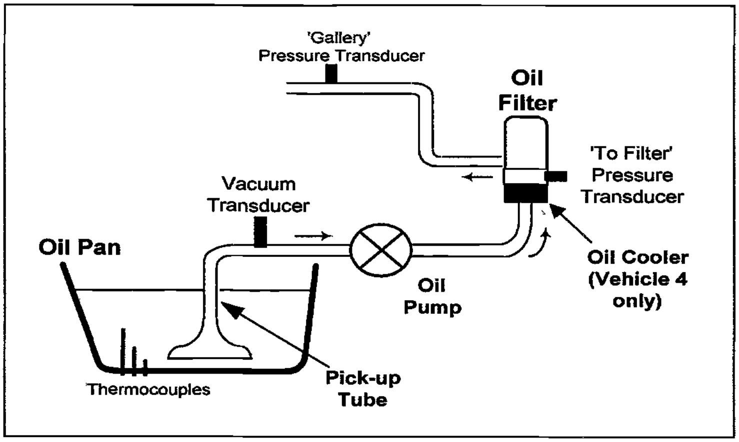


Figure 4 - *Schematic of Engine Instrumentation for Pumpability*

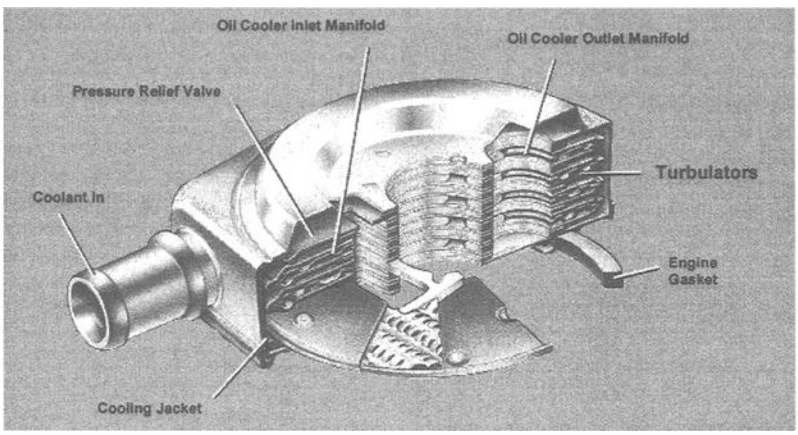


Figure 5 - *Oil Cooler (Vehicle 4, 4.6L V-8); Note Restricted Oil Flow (part length is approximately 10 cm)*

Oil Preparation

- Oil preheated for 90 minutes at 90°C prior to charging the engine; engine run briefly to circulate oil prior to cooldown.
- Double flush with double oil filter change when changing to a different oil. When testing the same oil in another cooldown cycle, single flush with oil filter change.

Cooling Cycle

The 24-hr cooling profile illustrated in Figure 6 was used for all AWCD work. Ambient air temperature was adjusted to ensure that oil pan temperatures followed a slow cooling profile (0.5°C/hr) through the gel index maxima observed for these oils (-23 to -24°C), with final test temperatures in the -33 to -38°C range (i.e. below minimum starting temperatures for these engines, as found in similar types in the ASTM LTEP studies on SAE 5W-30 oils [*9*]).

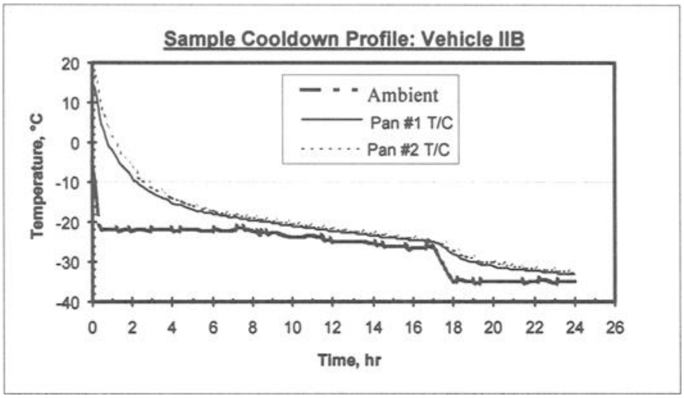


Figure 6 - *Cooling Profile for Pumpability Studies*

Pumpability Results

An example of a pumpability result on one of the Type I engines operating at -35°C ambient on Oil 'A' is shown in Figure 7. It is apparent that pressurization at the 'to filter' location (before the oil filter) and at the main gallery location occur rapidly for this oil.

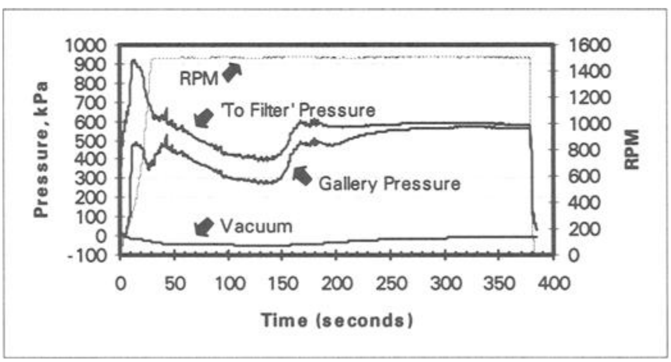


Figure 7 - *Pumpability Test on Oil 'D', -35°C Ambient, Car 1 (Vehicle Type I, 2.0L I-4)*

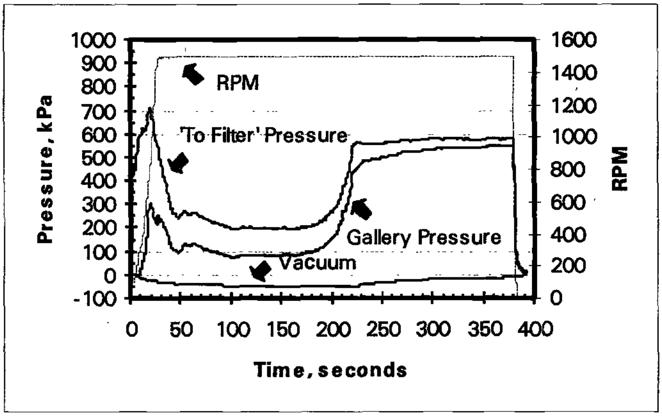


Figure 8 - *Pumpability Test on Oil 'D', -35°C Ambient, Car 1 (Vehicle Type I, 2.0L I-4)*

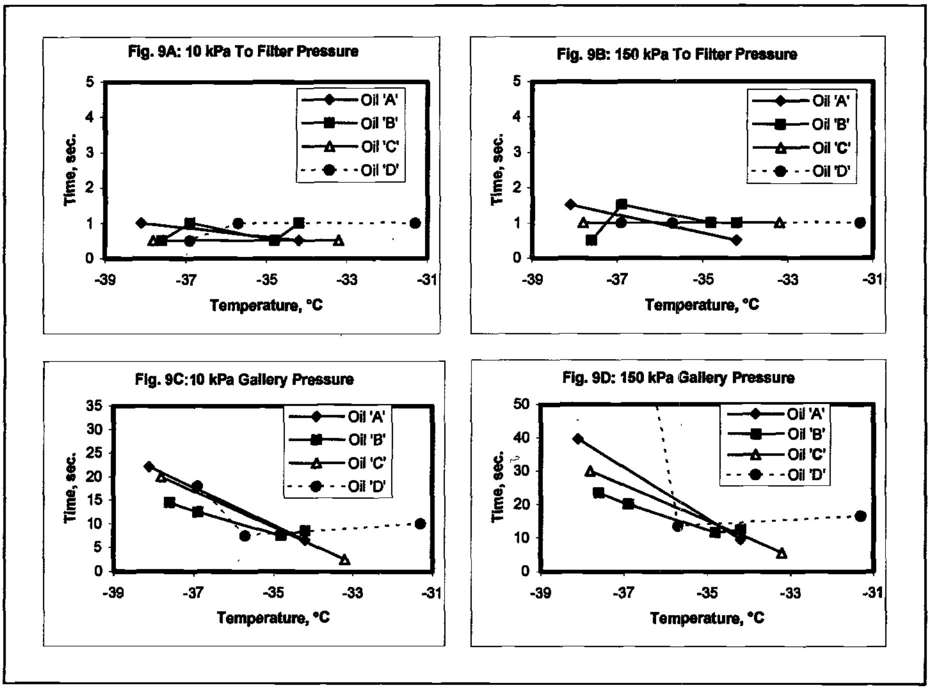


Figure 9 - *Summary of Temperature-Pressurization Data for Car 1 (2.0L I-4)*

In comparison, Figure 8 shows the results in the same vehicle/temperature combination with Oil 'D'. It should be noted that, even though this oil has a gel index less than one-half that of Oil 'A', its pressurization characteristics are not as good, consistent with the directionally poorer MRV viscosity.

Data from the pumpability tests on this engine were analysed in terms of time to reach 10 kPa or 150 kPa at the 'to filter' or main gallery location. These are illustrated as a function of oil sump temperature in the graphical figures associated with Figure 9. Pressurization times vs. temperature for Car 1 (2.0L I-4 engine) showed no effect of oil type if 'to filter' pressurization times were considered (Fig. 9A,B). Times to 150 kPa before the filter were all extremely low compared to one OEMs recommendation of 15 sec. max [*9*], even at the lowest temperatures. Times to pressurize the main gallery were somewhat longer, as expected for a 'down stream' location (Fig. 9C,D).

In this engine, Oil 'D' did not reach 150 kPa gallery pressure at the lowest temperature before the test was stopped. Recall that this 5W-30 had the lowest gel index value in the series, but the highest MRV viscosities. At the lowest test temperatures, *all* of the oils demonstrated the same phenomenon in this engine: rapid pressure rise in both 'to filter' and 'gallery' locations, followed by 'gallery' pressure dropping to near zero, while the 'to filter' pressure transducer continued to register 100-150 kPa (see Figure 10). These tests were terminated early to avoid engine damage - therefore gallery pressurization times at the lowest temperatures were not sustained values. This behaviour is suggestive of the 'air-binding' type of pumpability failures [*8*]. However, continued detection of pressure before the filter as well as vacuum readings at the vacuum transducer indicate that filter blockage is probably playing a role in downstream engine pressurization (one also wonders whether the original studies, with no vacuum measurement, would have labeled this as air-binding failure [*8*]). More work is needed to better understand this phenomenon, which was outside the scope of these tests.

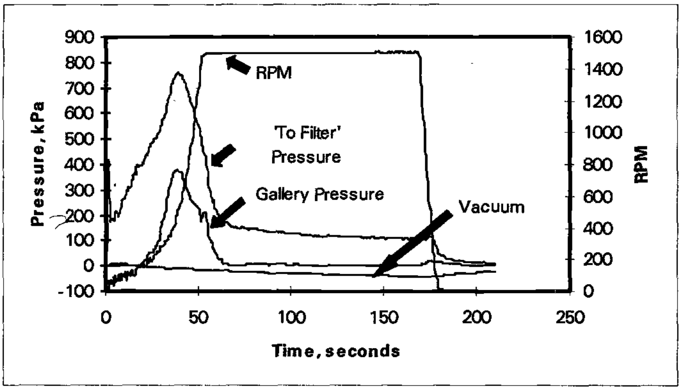


Figure 10 - *Pumpability Test on Oil 'C', -38°C Ambient, Car 1 (Vehicle Type I, 2.0L I-4)*

A similar graphical analysis for Car 2 (2.0L I-4 engine) is shown in Fig. 11A-D. As with Car 1, this engine showed rapid 'to filter' pressurization times in all cases. Gallery pressurization times were slightly better for Oil 'B' and 'D'. Again, gallery pressures at the lowest test temperatures were not sustained, regardless of gel index value.

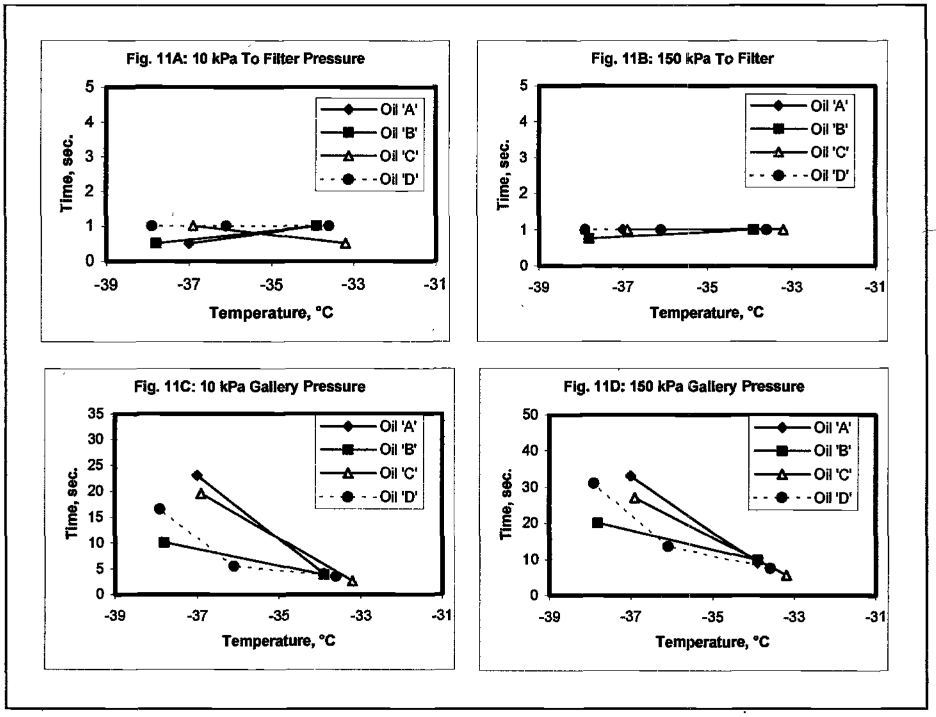


Figure 11 - *Summary of Temperature-Pressurization Data for Car 2 (2.0L I-4)*

All of the gallery pressurization data from Car 1 and 2 were compared to both estimated MRV viscosities[6] and Gel Index values for the test oils, as shown in Figures 12 and 13. It can be seen that the overall correlation appears to be much better for MRV viscosities than with gel index, and that, generally, the two vehicles are not dissimilar in their pressurization-viscosity behaviour.

[6] Interpolated/extrapolated from -35/-40°C D4684 results using the MacCoull-Walther-Wright equation.

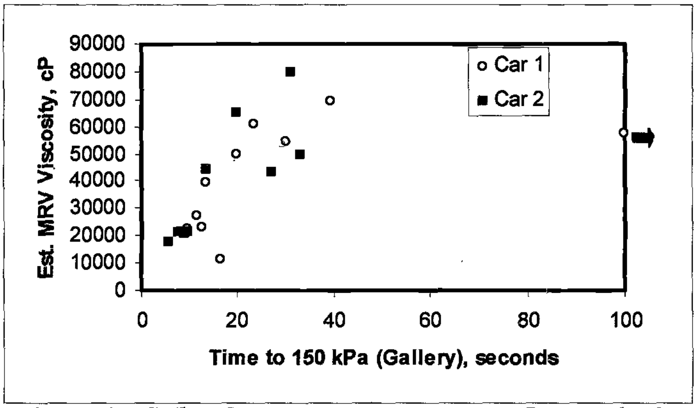


Figure 12 - *Gallery Pressurization Times versus Estimated MRV Viscosities for Cars 1 and 2*

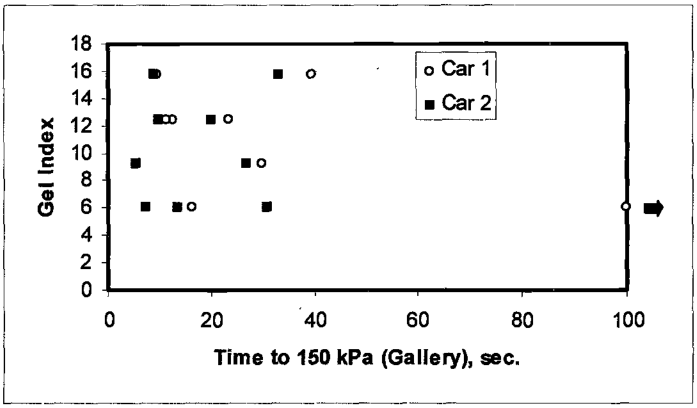


Figure 13 - *Gallery Pressurization Times versus Gel Index Values for Cars 1 and 2*

Summary results from Car 3 (Type IIA, 4.6L V-8) are shown in Figure 14. 'To filter' pressurization times to 10 kPa showed no differences among the oils (Fig. 14A), although the time to 150 kPa was longer for Oil 'A' (Fig. 14B). In terms of gallery pressurization times, Oil 'A' took longer to reach 10 kPa, but Oil 'D' had the longest times to reach 150 kPa (Fig. 14C,D). No evidence of air-binding pumpability characteristics was observed with any of these runs; this is not unexpected, given the relatively high oil height and large L/D^4 ratio for this engine (see Table 3).

Although Car 4 was equipped with the same 4.6L V-8 engine as Car 3, pumpability characteristics were very different, as evidenced by the time-pressure traces of Oil 'C' at -35°C ambient (compare Figures 15 and 16).

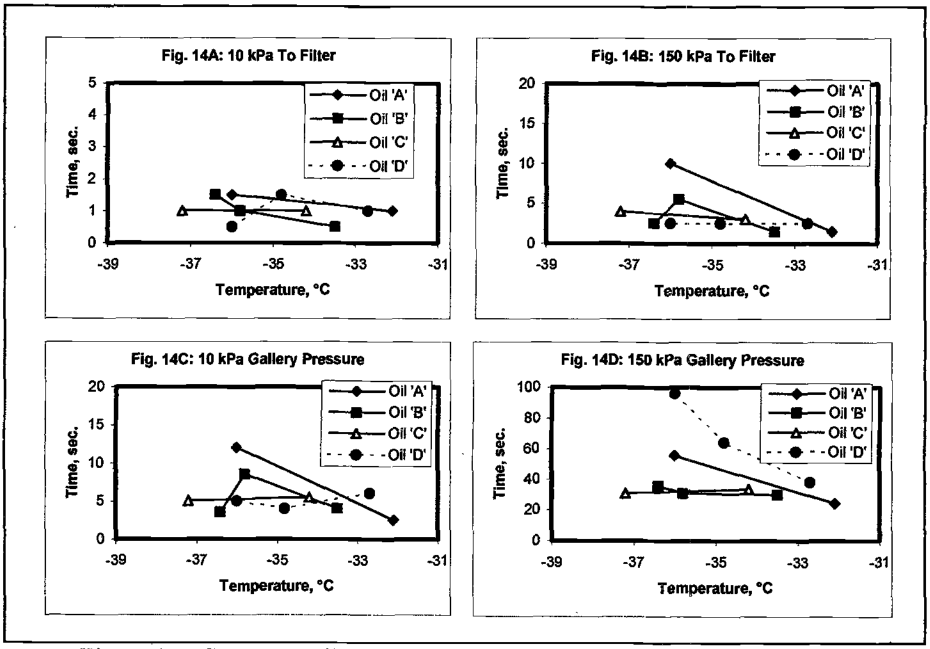


Figure 14 - *Summary of Temperature-Pressurization Data for Car 3 (4.6L V-8)*

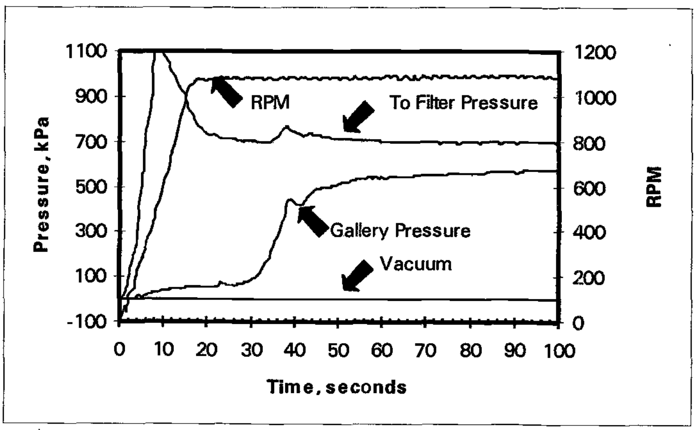


Figure 15 - *Pressure-Time Traces for Oil 'C' in Vehicle 3 at -35°C*

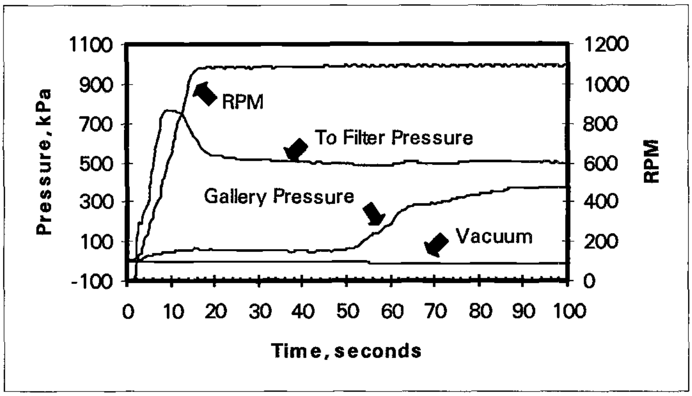


Figure 16 - *Pressure-Time Traces for Oil 'C' in Vehicle 4 at -35°C*

With identical oil temperatures in the two runs, (a) oil pressure developed more slowly in Vehicle 4 than in Vehicle 3, particularly at the main gallery location, and (b) the equilibrium pressure values at both transducer locations were approximately 200 kPa lower for Vehicle 4, equipped with the oil cooler. Clearly, the presence of the oil cooler, installed before the filter (see Fig. 4), restricts the flow of the cold oil thereby producing significant pressure drop (~200 kPa) and leading to increased pressurization times at downstream locations. This is apparent if times to 150 kPa gallery pressure for the 2 engines are plotted against estimated MRV viscosities for the test oils (Fig. 17); there are clearly two populations of data, and beyond an MRV viscosity of ~45,000 cP (obtained at the lowest test temperatures), pressurization times in Car 4 become extremely long.

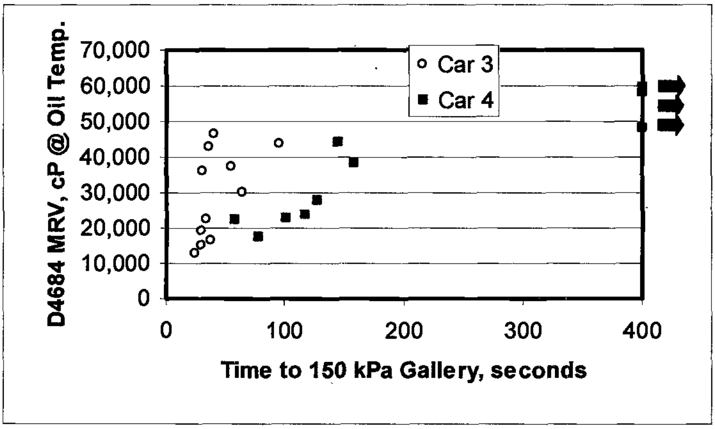


Figure 17 - *Comparison of Pressurization Times vs Estimated MRV Viscosities for Vehicles 3 and 4 (4.6L V-8) Engines*

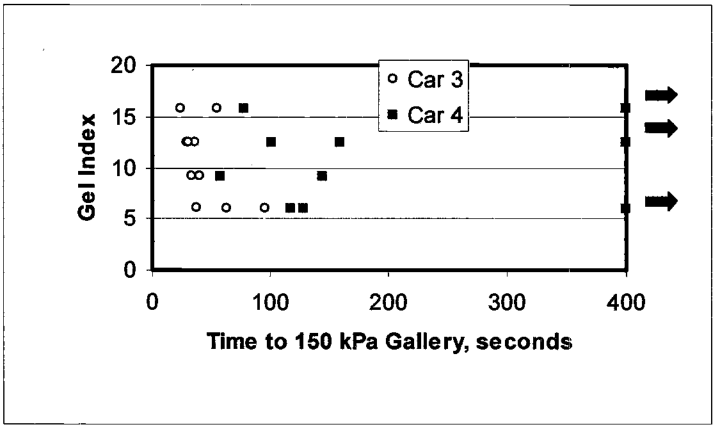


Figure 18 - *Comparison of Pressurization Times vs Gel Index for Vehicles 3 and 4 (4.6L V-8) Engines*

A similar plot of gel index versus pressurization times (Figure 18) also indicates longer pressurization with Car 4 vs. Car 3 for a given GI. Gel index itself, however, shows no correlation to pressurization times.

As mentioned earlier in this paper, the two engine types selected for this study encompassed very different designs if L/D^4 and oil height is considered. These are illustrated in the plot shown in Figure 19 and compared to those covering the original 1975 ASTM engine pumpability program where both flow-limited and air-binding behaviour was observed [*8*].

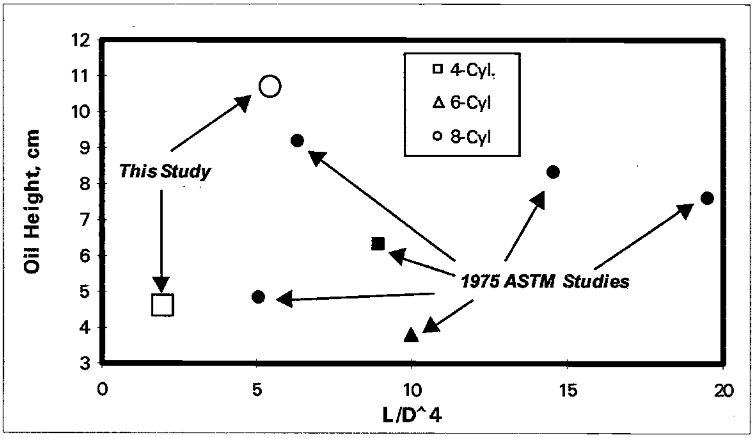


Figure 19 - *Comparison of Oil Pick-Up Tube Length ÷ Diameter^4 versus Oil Height; Current Study and Original ASTM Study [8]*

Flow-limited behaviour would be expected in engines equipped with long, small diameter pick-up tubes (i.e. high L/D^4 ratios), and large oil heights, while air-binding behaviour should be enhanced by shorter, larger diameter tubes and low oil heights[7]. This is somewhat borne out by the observation that the 1975 vintage 8-cylinder engine with the highest L/D^4 ratio and relatively generous oil height showed no air-binding pumpability failures [*8*]. Compared to these older engines, the models selected for this study had comparable or lower L/D^4 ratio. Of the two selected, the V-8 design would be expected to have less air-binding tendencies, and certainly none of the tests conducted even hinted at 'pseudo air-binding' behaviour (i.e. gallery pressurization was associated only with a steady rise to an equilibrium value with continued running as in Figure 15). The 2.0L I-4 engine, however, with its low L/D^4 ratio and low oil height (the latter in the same range as some of the earlier engine types), would be expected to be more susceptible to air-binding, and if only gallery pressurization at very low temperatures is considered, it would appear to be encountered in these tests. As discussed earlier, though, this pumpability limitation is not consistent with vacuum readings before the pump or 'to filter' pressure measurements. Of course, the oils used in this study were well-behaved with respect to yield stress, while oils tested in the 1975 ASTM report covered formulations at much higher yield stress values [*8*]. In addition, the earlier study did not rely on vacuum readings before the pump, thereby raising unanswerably questions regarding the true nature of some of the failures reported as air-binding.

We were also interested in measuring the rheological properties of some of the test oils obtained after the pumpability experiment was conducted, to see if any change in lubricant rheology had occurred after this very short running time. As shown in Table 4, several of the drains from vehicles operating on Oil 'A' and Oil 'B' (those with the highest fresh oil gel index, see Table 2), showed substantial decreases in the GI. This was not, however, the case for the D4684 MRV viscosities (measured at -35°C), which showed relatively modest changes from their fresh oil values. This suggests to the authors that the gel index may be very sensitive to structures in the oil which are readily destroyed during even brief operation in the engine.

Table 4 - *Rheology of Used Oils from Pumpability Tests*

		Used Oil Result from Drain Sample (by Car)			
Test Oil	**Measurement**	**Car 1**	**Car 2**	**Car 3**	**Car 4**
Oil 'A'	Gel Index	12.0	7.5	5.8	7.6
	-35°C TP-1, cP	27,050	26,000	27,400	24,800
	KV100, cSt	10.58	10.46	10.77	10.50
Oil 'B'	Gel Index	7.0	5.2	5.0	9.5
	-35°C TP-1, cP	25,600	25,700	24,600	24,400
	KV100, cSt	10.51	10.55	10.69	10.44

[7] This is an oversimplified analysis, since pick-up tube number and degree of bends may have other effects [*8*].

Conclusions

(1) Full-scale pumpability testing (2.0L I-4 and 4.6L V-8 engines) using a modified slow-cooling profile on four oils ranging from 15.7 to 6.0 gel index did not show an effect of GI on either pressurization times or tendencies to undergo air-binding failure. Correlation of 150 kPa Gallery pressure times to MRV viscosities was observed, which depended on engine type.

(2) One engine type (2.0L I-4) with relatively low L/D^4 ratio and oil height showed 'pseudo air-binding' behaviour at the very lowest test temperatures with all oils. Further analysis of the vacuum reading before the pump, and of the gallery pressure suggests another mechanism rather than true air-binding failure. There was no correlation between the observation of this phenomenon and the gelation index of the oil.

(3) Engine design and hardware differences can have profound influences on low temperature pumpability. This was particularly evident in the low temperature comparison of two 4.6L V-8 engines, in which the one equipped with a multi-pass oil cooler showed significantly longer pressurization times under the same conditions.

(4) Several drain samples from pumpability tests on the higher gelation index oils (15.7, 12.4) showed reduced GI values compared with those for the fresh oils, although -35°C MRV viscosities changes were relatively small. This suggests that even small amounts of engine operation may affect the very subtle low temperature oil structures that the Scanning Brookfield technique appears to detect.

References

[1] Henderson, K. O., Manning, R. E., May, C. J. and Rhodes, R. B., "New Mini-Rotary Viscometer Temperature Profiles That Predict Engine Oil Pumpability," SAE Paper No. 850443.

[2] Selby, T. W. and Piasecki, D. A., "Low Shear, Low Temperature Properties of Engine Oils - Techniques with the Brookfield Viscometer," SAE Paper No. 800367.

[3] Selby, T. W., "The Scanning Brookfield Technique of Low-Temperature, Low-Shear Rheology - Its Inception, Development, and Applications," *Low Temperature Lubricant Rheology Measurement and Relevance to Engine Operation, ASTM STP 1143*, R.B. Rhodes, Ed., American Society for Testing and Materials, West Conshohoken, PA, 1992, pp. 33-64.

[4] Selby, T. W., "Problems in Bench Test Prediction of Engine Oil Performance at Low Temperature," SAE Paper No. 922287.

[5] Selby, T. W., "The Use of the Scanning Brookfield Technique to Study the Critical Degree of Gelation of Lubricants at Low Temperatures," SAE Paper No. 910746.

[6] Engine Oil Licensing and Certification System, API 1509, 14th Edition, December, 1996, pg D-6.

[7] "Low-Temperature Properties of Engine Oils," Lubrizol NewsLine, Vol. 16, No. 1, February, 1998.

[8] ASTM DS-57, "*Low Temperature Pumpability Characteristics of Engine Oils in Full Scale Engines,*" American Society for Testing and Materials, Philadelphia, PA 19103, September 1975.
[9] *Cold Starting and Pumpability Studies in Modern Engines, ASTM Research Report RR-D02-1442*, American Society for Testing and Materials, West Conshohocken, PA, November 1998.
[10] May, C. J., Koenitzer, B. A. and Lai, P. K. S. L, "A Study of Gel Index and MRV Effects on SAE 5W-30 Pumpability," SAE Paper No. 982641.

ISBN 0-8031-2857-6