SUMMARY OVERVIEW OF INTERNATIONAL CONFERENCE ON LONG-TERM STORAGE STABILIZATION (U) SOUTHWEST RESEARCH INST SAN ANTONIO TX BELVOIR FUELS AND LUBR. L L STAVINOGHA UNCLASSIFIED OCT 86 BFLRF-220 DAAK70-85-C-0007 F/G 21/4 NL
SUMMARY OVERVIEW OF SECOND INTERNATIONAL CONFERENCE ON LONG-TERM STORAGE STABILITIES OF LIQUID FUELS

SPECIAL REPORT
BFLRF No. 220

L. L. Stavinoha
Belvoir Fuels and Lubricants Research Facility (SwRI)
Southwest Research Institute
San Antonio, Texas

Under Contract to
U.S. Army Belvoir Research, Development and Engineering Center
Materials, Fuels and Lubricants Laboratory
Fort Belvoir, Virginia

Contract No. DAAK70-85-C-0007

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October 1986
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| 19. ABSTRACT (Continue on reverse if necessary and identify by block number) | The 2nd International "Conference on the Long-Term Storage Stabilities of Liquid Fuels" was held July 29 - August 1, 1986 in San Antonio, Texas at the Hilton Palacio Del Rio Hotel. The conference was hosted by Southwest Research Institute (SwRI) and sponsored in principle by the United States Department of Energy and the United States Department of Defense.

The many papers and representatives of the U.S. Army and U.S. Navy helped make this conference technically broad based. A strong conference program was realized by the support and participation of many other Military Defense Establishments (i.e., outside the U.S.). This, combined with strong industrial participation, produced an overall successful conference.

The Conference program had 67 papers in 10 sessions, including a number of panel presentations covering Strategic and Emergency Fuel Storage; Operational Storage and Fuel Systems; Fuel Biocides | |
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19. ABSTRACT (Cont'd)

and Microbiology; Methods for Characterization, Analysis, and Testing; Filtration; Theoretical Aspects of Fuel Stability; Test Method Development and Evaluation; Shale/Coal Liquids and Additive Chemistry; and the Effect of Refinery Processing and Additives on Stability.

This conference discussed liquid hydrocarbon fuels with respect to their quality and resistance to change in storage. The liquid fuels included gasoline, middle distillate (diesel, turbine, jet, and burner), residual fuel oil, and crude oil. The increasing importance of this subject continues to be evidenced by considerable research effort in many countries. In keeping with the spirit of the first conference held in Tel Aviv, Israel in 1983, pooling of unclassified knowledge during this conference proved to be of benefit to all participants.

Of the 202 conference attendees, 70 were from 21 countries other than the United States. The technical input provided from investigators from other countries proved to be most enlightening to the U.S. research community present at the conference.

Overall observations included:

- In general, jet fuel long-term storage stability appears satisfactory. Heavily hydrotreated stocks require the addition of oxidation inhibitors, primarily hindered phenols, to prevent peroxide formation.

- The storage stability of middle distillates, such as gas oils, diesel fuels and heating oils, depends upon the amount and type of cracked stocks included. Amine-type stabilizers, as opposed to primary amine-type antioxidants, appear most successful in preventing sediment formation, but the selection of additives still seems to be pragmatic and empirical rather than based on theory.

- Although sediment weight is used by most as a criterion of fuel suitability, several investigators emphasized the lack of correlation between sediment weight and fuel filtration characteristics. The ability of particles to settle or to stay suspended as a function of particle size appears to be a major factor. Several laboratory filtration tests are under development.

- The need for a short-term test for the prediction of storage stability continues, but no test which accelerates conditions by increasing test temperature is dependable over a wide range of fuels. In this connection, several investigations using oxidation accelerators at lower temperatures appear worthy of further study.

- Microorganisms can cause storage difficulties, particularly in wet caverns. However, available biocides can control the problems, except for wet cavern storage. Some biocides were reported to be effective at low parts per million concentrations in fuels.

- Several new, useful procedures are available to detect the onset of fuel corrosivity by techniques more sensitive than the standard corrosion tests.

- A new device for measuring lacquer-type deposits on thermal oxidative test tubes (ASTM D 3241) uses the principal of dielectric breakdown voltage. This method appears to be most useful in fuel thermal stability programs that are examining neat and storage changes as well as additive and metallurgical effects on deposit kinetics.

- New techniques utilizing mass spectroscopy and supercritical liquid chromatography are helping to understand the mechanism of sediment formation in middle distillate fuels.
FOREWORD

The 2nd International "Conference on the Long-Term Storage Stabilities of Liquid Fuels" was held July 29 - August 1, 1986 in San Antonio, Texas at the Hilton Palacio Del Rio Hotel. The conference was hosted by Southwest Research Institute (SwRI) and sponsored in principle by the United States Departments of Energy and Defense. Financing of the Conference was provided for through a registration fee, additional sales of Conference proceedings, and SwRI sponsorship.

This conference discussed liquid fuels with respect to their quality and resistance to change in storage. The liquid fuels included gasoline, middle distillate (diesel, turbine, jet, and burner), residual fuel oil, and crude oil.

The Conference program encompassed 67 papers in 10 sessions, including a number of panel and poster presentations:

SESSION 1: Fuel Surveys and Long-Term Storage Studies
Panel: Quality of Fuels in Storage
SESSION 2: Strategic and Emergency Fuel Storage
SESSION 3: Operational Storage and Fuel Systems
SESSION 4: Microbial Aspects of Fuel Stability
SESSION 5: Biocides and Microbiology of Fuels
Panel: Biocides for Liquid Fuels
SESSION 6: Methods for Characterization, Analysis, and Testing
Panel: Filtration
SESSION 7: Theoretical Aspects of Fuel Stability
SESSION 8: Test Method Development and Evaluation
Panel: Accelerated Stability Test Error Sources
SESSION 9: Shale/Coal Liquids and Additive Chemistry
SESSION 10: Effect of Refinery Processing and Additives on Stability
Panel: Refinery Processing and Additives for Diesel and Naval Distillate

The subject of long-term storage stabilities of fuels is complex, and the scope is best illustrated by the session topics. The increasing importance of this subject continues to be evidenced by considerable research effort in many countries. In keeping with the
spirit of the first conference held in Tel Aviv, Israel in 1983, pooling of unclassified knowledge during this conference proved to be of benefit to all participants.

The conference proceedings include most of the papers presented at the conference in the order they were presented in each of the sessions. Chairmen for each of the sessions are acknowledged in the Conference Program.

The Organizing Committee of this second International Conference on Long-Term Storage Stabilities of Liquid Fuels wishes to express its sincere thanks and appreciation to the various authors who have supported and who have contributed to the success of this Conference.

It is very necessary to recognize the many papers and representatives of the U.S. Army and U.S. Navy which helped make this conference technically broad based. A strong conference program was realized by the support and participation of many other Military Defense Establishments (i.e., outside the U.S.). This, combined with strong industrial participation, produced an overall successful conference.

This summary overview was prepared and distributed by Belvoir Fuels and Lubricants Research Facility (BFLRF), at Southwest Research Institute, San Antonio, TX, under Contract No. DAAK70-85-C-0007 and covers the period May 1986 to October 1986. Contractor's representative was Mr. F.W. Schaekel, Materials, Fuels, and Lubricants Laboratory/STRBE-VF, U.S. Army Belvoir Research, Development and Engineering Center, Fort Belvoir, VA.
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I. CONFERENCE PROGRAM

The increasing importance of the long-term storage stability of liquid fuels continues to be evidenced by considerable research effort in many countries. The purpose of this conference was to bring researchers together to discuss liquid fuels with respect to their quality and resistance to change. The liquid fuels included gasoline, middle distillate (diesel, turbine, jet, and burner), residual fuel oil, and crude oil. The subject of long-term storage stabilities of fuels is complex, and the scope is best illustrated by the session topics and presentation titles provided in the following conference program:

- Keynote Speech: Dr. Robert N. Hazlett
  U.S. Naval Research Laboratory

- SESSION 1: FUEL SURVEYS AND LONG-TERM STORAGE STUDIES
  Chairmen: Wayne Vreatt¹
  Amos Ishai²

  • Storage Stability of Residual Fuel Oils [1]†
    National Institute for Petroleum and Energy Research

  • Long-Term Storage Stabilities of Crude Oil Reserved by Japan National
    Oil Corporation [24]
    T. Hara
    Yokohama National University

  • Test Methods and Experience on Long-Term Storage Stabilities of
    Gasolines and Middle Distillates in Switzerland [19]
    E. Gartenmann
    Swiss Federal Institute of Test Materials (EMPA)

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- Highlights of "Field Tests on Storage Stability of Gasoline, Jet Fuel, and
  Gas Oil" [41]
  D. Luria
  Israel Fuel Authority

- Stability Measurements of Commercial Marine Fuels From a Worldwide
  Survey [25]
  D.R. Hardy, et al.
  U.S. Naval Research Laboratory

- A Recent Instability Occurrence With Naval Distillate NATO F-76 [21]
  R. Giannini, H. Modetz*
  David W. Taylor Naval Ship Research and Development Center
  * Accurex Corporation

¹ Office of the Chief of Naval Research, Attn: OCNR-1263, Wayne Vreatt, 800 Quincy
  St., Arlington, VA 22217-5000
² Paz Oil Company Ltd., Attn: Amos Ishai, P.O. Box 434, Haifa, Israel 31003
† Numbers in brackets refer to paper number as identified in Conference Proceedings.
SESSION 2: STRATEGIC AND EMERGENCY FUEL STORAGE

Chairmen: Harry N. Giles
           Elliott Katz

- Storage Stability of a Kerosene and a Jet Fuel [39]
  N. Li, J.R. Tzou, H. Chang, and S.M. Wang
  National Tsing Hua University

- Application of a Field Fuel Quality Monitor to Surveillance of
  Prepositioned Fuel Stocks [74]
  S.R. Westbrook, L.L. Stavinoha, J.G. Barbee, L.L. Bundy, and J.V.
  Mengenheuser*
  Belvoir Fuels and Lubricants Research Facility (SwRI)
  * Belvoir Research, Development and Engineering Center

- A Quality Control System for Diesel Fuel in Long-Term Storage [67]
  K.H. Strauss
  Consultant, Petroleum Fuels

FILM:

- In-Ground Oil Storage Containers "From Whence It Came" [69]
  J.E. Thrasher, C.S. Dunn
  Fenix and Seisson, Inc.

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Chairman: Ron Layne
           Lawrence Long

- Effects of Polymeric Coating Systems for Concrete Fuel Tanks on the
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  B. Polishook, Y. Geva*, and R. Fass*
  Petroleum, Ltd.
  * The Israel Institute for Biological Research

- Consequences of Sulphur Compound Conversions in Storage of Jet Fuels
  [4]
  J.B. Asher, N. Por*, and A.B. Shavit**
  Israel Institute of Petroleum and Energy
  * Oil Refineries Ltd.
  ** Delek, The Israel Fuel Corp., Ltd.

- Stability Properties and Compatibilities of Residual Fuels [57]
  N. Por, R. Brauch, and N. Brodsky
  Oil Refineries Ltd.

3 U.S. Department of Energy, FE-422, 3G-029 Forrestal Building, Washington, DC 20585
4 Principal Director, The Aerospace Corporation, 2350 E. El Segundo Blvd., El Segundo,
  CA 92357
5 Naval Sea Systems Command (NAVSEA), Attn: SEA 05M4, Washington, DC 20382
6 Navy Petroleum Office (NPO), Cameron Station 8B427, Alexandria, VA 22304-6180
- Stability Properties and Compatibilities of Residual Fuels [57]
  N. Por, R. Brauch, and N. Brodsky
  Oil Refineries Ltd.

- Operational Problems With Marine Fuel Oils [77]
  M.F. Winkler
  Seaworthy Systems, Inc.

- **SESSION 4: MICROBIAL ASPECTS OF FUEL STABILITY**

  **Chairmen:** Rex Neihof\(^7\)
  R. Fass\(^8\)

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    U.S. Naval Research Laboratory

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  - Microbiological Studies in Rock Caverns With Jet Fuel, Heavy Fuel Oil and Crude Oil [49]
    A. Norqvist, R. Roffey, and A. Edlund
    National Defence Research Institute, Sweden

  - Effect of Microbial Contamination in Storage Tanks on the Long-Term Stability of Jet Fuel [16]
    R. Fass, J.B. Asher*, and A.B. Shavit**
    Israel Institute for Biological Research
    * The Israel Institute of Petroleum and Energy
    ** Delek, The Israel Fuel Corp., Ltd.

  - Methods to Monitor Biodeterioration of Jet Fuel During Long-Term Storage in Rock Caverns [61]
    R. Roffey, A. Norqvist, and A. Edlund
    National Defence Research Institute, Sweden

  - Update on Fuel Treatment Program for Middle Distillate Long-Term Fuel Storage and Storage Tank Corrosion [13]
    H.L. Chesneau
    Fuel Quality Services, Inc.

\(^7\) Code 6181, U.S. Naval Research Laboratory, Chemistry Division, Washington, DC 20375-5000
\(^8\) Israel Institute for Biological Research, P.O. Box 19, Ness-Ziona, Israel 70450
- Effect of Microbial Contaminants on the Corrosion of Fuel Storage Tanks [60]
  Faculty of Veterinary, UNLP
* Research and Development Branch, Y.P.F.
** Bioelectrochemistry Section, INIFTA, Argentine

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  K. Bosecker, M. Teschner, and H. Wehner
  Bundesanstalt fur Geowissenschaften und Rohstoffe (Abstract Only)

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  Belvoir Fuels and Lubricants Research Facility (SwRI)
* Belvoir Research, Development and Engineering Center

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* Seatech Investigation Services Ltd.
** Dalhousie University

9 Code 6181, U.S. Naval Research Laboratory, Chemistry Division, Washington, DC 20375-5000
10 Delek, Israel Fuel Corporation, 6 Prof Kaufman St. 61018, P.O.B. 50250, Tel Aviv, Israel 61500
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[11] Southwest Research Institute, 6220 Culebra, P.O. Drawer 28510, San Antonio, TX 78284
[12] NIPER, P.O. Box 2128, Bartlesville, OK 74005
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    * Department of Defence, Australia

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    U.S. Naval Research Laboratory
    * Department of Defence, Australia

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13 Head, Fuels Section, U.S. Naval Research Laboratory, Code 6180, Washington, DC 20375-3559
14 David Taylor Naval Ship Research and Development Center, Code 2832, Annapolis, MD 21402
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  * Belvoir Research, Development and Engineering Center

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15 Commander, U.S. Army Belvoir Research, Development and Engineering Center,
  Attn: STRBE-VF, Fort Belvoir, VA 22060-5606
16 Royal Aircraft Establishment (UK), RAE (Cobham) Fairmile, Cobham, Surrey,
  KT11 1BJ, United Kingdom
17 Fuels and Lubricants Lab., National Research Council of Canada, Ottawa Ontario,
  Canada  K1A OR6
- **Effect of Nitrogen and Sulphur Compounds on the Deterioration of Fuels at Elevated Temperatures [59]**
  K.T. Reddy, N.P. Cernansky, and R.S. Cohen*
  Drexel University
  * Temple University

- **The Influence of Polar Compounds on the Stability of Jet Fuels [84]**
  D.R. Kendall, P.A. Stevenson
  Shell Research Ltd.

- **Thermal Stability of Diesel Fuels [85]**
  L.L. Stavinoha, J.G. Barbee, D.M. Yost
  Belvoir Fuels and Lubricants Research Facility (SwRI)

- **Effects of an Unstable Diesel Fuel on Injector Coking and Vehicle Performance [86]**
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  General Motors Research Labs

**SESSION 9: SHALE/COAL LIQUIDS AND ADDITIVE CHEMISTRY**
(Concurrent with Session 8)

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Nahum Por19

- **Introductory Remarks:** Art Hartstein [87]
  U.S. Department of Energy, USA

- **Influences of Sulphur, Nitrogen, and Oxygen Bearing Compounds on Diesel Fuel Storage Stability [46]**
  R.E. Morris
  U.S. Naval Research Laboratory

- **Effects of Shale-Derived Polar Compounds on Diesel Fuel Stability [15]**
  J.V. Cooney, E.J. Beal, G.W. Mushrush, and R.N. Hazlett
  U.S. Naval Research Laboratory

- **Storage Stability of Coal-Derived Liquids [34]**
  B. Hoesterey, W.H. McClennen, G.R. Hill, and H.L.C. Meuzelaar
  University of Utah

- **Fates of Heteroatoms When Processing Synthetic Crudes [68]**
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  University of Tulsa

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  N. Por, N. Brodsky, D. Givoni*, and A. Raweh*
  Oil Refineries, Ltd.
  * PAMA, Energy Sources Development

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18 U.S. Department of Energy, Deputy Director, M.S. D-122, Washington, DC 20545
19 Oil Refineries Ltd., P.O. Box 4, Haifa, Israel 31000
• The Role of Antioxidants in Improving Stability Properties of Shale Oil and Its Products [52]
  N. Por, N. Brodsky, D. Givoni*, and A. Raweh*
  Oil Refineries, Ltd.
  * PAMA, Energy Sources Development

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- Hydrotreatment of Light Cycle Oil for Stabilization of Automotive Diesel Fuel [50]
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- Causes of Color Changes and Particulate Formation in Navy Distillate Fuels [10]
  D.W. Brinkman, O. Bhan, and B. Carley*
  National Institute for Petroleum and Energy Research
  * Defense Fuel Supply Center
II. OVERVIEW SUMMARY

The following summary provides an overview of the Conference. The actual papers presented at the conference are available in the Proceedings of the Conference*.

SESSION 1: FUEL SURVEYS AND LONG-TERM STORAGE STUDIES

Anderson [1]** described the validation of several rapid tests to identify instability and incompatibility problems in residual fuels. Commonly used tests are considered inadequate, but coke content (toluene-insoluble material) and the dry sludge test developed by Shell appear promising.

Hara [24] studied the stability of four crude oils in large volume storage in Japan. Extensive structural analyses of both crudes and sludges indicate acidic nitrogen compounds to be responsible for sludge formation in one crude, but lack of instability in other crudes precludes prediction of instability at this time.

Gartenmann [19] has monitored Swiss motor gasoline and diesel fuel in above- and below-ground tank storage for 10 years and more. Property limits for procurement and for continued storage are given. Prediction of additional storage life is based on periodic bottle tests run at 40°C for gasoline and 50°C for diesel fuel.

By comparing drum to floating roof tank storage, Luria [41] hopes to use drum storage results to identify causes for gasoline, jet fuel, and gas oil instability. Storage periods exceeding 5 years were possible with properly inhibited fuels made at two Israeli refineries. (Apparently cyclic sediment formation was attributed by other attendees to periodic sediment precipitation).

Hardy [25] considered sediment formed at 43°C after 12 weeks, at 80°C after 2 weeks, and at 95°C for 16 hours (ASTM D 2274) to be equivalent for 36 commercial marine distillate fuels (collected in a worldwide survey). Stability of the marine gas oils was*

* Proceedings of the 2nd International Conference on Long-Term Storage Stabilities of Liquid Fuels, edited by Leo L. Stavinoha and published by Southwest Research Institute, P.O. Drawer 28510, San Antonio, TX 78284, October 1986
** Numbers in brackets refer to the paper number in the Conference Program.
best, with marine diesels the poorest. Fuels from the Indian Ocean area showed generally high sediment formation in these tests.

Giannini [21] reported on additive blending to correct high sediment formation in a batch of naval distillate (NATO F-76). A commercial amine-type additive showed some improvement, but the current specification, MIL-F-16884H, is considered inadequate to preclude fuels with poor stability. (Comments indicated that earlier additive blending at the refinery might have prevented the problem.)

SESSION 2: STRATEGIC AND EMERGENCY FUEL STORAGE

Li [39] added phenols and copper powder to a Taiwanese JP-5 to accelerate degradation which was measured by various sophisticated techniques. Light scattering showed particle formation at an earlier stage than conventional sediment-measuring techniques. Fuel copper content after removal of the copper powder and before the onset of sediment formation is also proposed for predicting instability.

Westbrook [74] described a field fuel quality monitor which permits onsite measurement of stored fuel quality. Sediment is measured by light absorption of a filter membrane, while further storage life is estimated by an automated 150°C test. The unit has been successfully used in several field studies.

Strauss [67] outlined a quality control system for diesel fuel in long-term storage for critical applications. The tests required of the supplier and the user are described and the reasons for the tests and their recommended limits are given.

Thrasher [69], in a documentary film "From Whence It Came" showed the construction from start to finish of a huge concrete storage vessel in South Africa.

SESSION 3: OPERATIONAL STORAGE AND FUEL SYSTEMS

Polishook [51] examined four polymeric lining systems for concrete tanks for their effects on jet fuel and the fuel's effect on the polymers, with particular emphasis on microbial contamination. One system, SN-7012, was judged most suitable.
Asher [4] tested the effect of sulfur content and composition on copper and silver corrosion. The inhibition by sulfides and disulfides of corrosive fuel containing up to 6 ppm elemental sulfur was reported. Fuels containing essentially no sulfur became corrosive to copper due to peroxide formation.

Por [57] evaluated various residual fuel inspection tests to develop controls over visbreaker severity. For satisfactory residual blend stability, fuel peptization values should be greater than 2 and storage temperatures should be as low as possible. Reduction of oxygen contact with the fuel would also be helpful. (Others in the audience questioned complete reliance on peptization number and recommended the additional use of xylene equivalence for visbreaker control.)

Winkler [77] presented a general review of problems noted with marine residual fuels due to heavy cracking or visbreaking. Compatibility and stability were cited as the major problems with inadequate blending being a frequent culprit.

SESSION 4: MICROBIAL ASPECTS OF FUEL STABILITY

Neihof [48] described U.S. Navy experience with microorganisms, starting with sulfate-reducing bacteria and later with fungi which caused wing tank corrosion. No major problems exist today. From a Navy standpoint, water-soluble additives appear to be the most effective way to minimize microbial growth.

Fass [17] stored jet and diesel fuels in rock models simulating wet cavern storage. Jet fuel showed no adverse effects after 5 years of static storage; diesel none after 1 year to date. Accelerated growth conditions caused the jet fuel to go off-specification on WSIM and on silver and copper corrosion due to the action of sulfate reducers.

Norqvist [49] described microbial problems in Swedish wet rock cavern storage of JP-4 jet fuel, crude oil, and heavy residual fuel. Hydrogen sulfide has been produced in jet fuel caverns located near the sea. Methane is being formed in the heated residual fuel storage. Solutions include minimizing sea water contact, fuel-water interface area, and water leakage into the caverns.
Fass [16] studied microorganisms in the laboratory and in storage tanks over a 5-year period. Degradation of WSIM and silver corrosion occurred in one tank which was improved by removal of the bottom fuel layer. Five-year storage of uninhibited jet fuel is feasible under close monitoring and fuel bottom removal when necessary.

To detect incipient silver corrosion, Roffey [61] developed a rapid, sensitive method based on piezoelectric crystals. A new method for elemental sulfur was also described. Radioactive-labelled compounds are being used to measure biological activity.

Chesneau [13] gave a general discussion of fuel treatment with the additive package described in MIL-S-53021 which includes an antioxidant, a metal deactivator, a dispersant, a corrosion inhibitor, and a biocide. Storage tank corrosion/leakage was also vividly described through a large number of slides.

Reinoso [60] studied the electrochemical behavior of SAE 1020 steel and 2024 aluminum alloy to estimate the corrosion hazard in storage tanks due to microbial contaminants in jet fuel. In the presence of metabolic products, a breakdown of passivity occurs, leading to localized corrosion.

Bosecker [8] provided an abstract on in-vitro biodegradation of crude oils in which parameters important for the course of the reaction were identified and optimized.

SESSION 5: BIOCIDES AND MICROBIOLOGY OF FUELS

Holmes [81] provided an introduction to the types of microorganisms found and types of problems caused in fuel oil systems. Physical and chemical means to controlling degradation and fouling of fuel oil systems were presented.

Andrykovitch [2] evaluated five different biocides for effectiveness in controlling growth of major fungal contaminants, retention of effectiveness with storage time in fuel/water systems, and susceptibility to inactivation by fuel tank sludge. A mixture of isothiazolin compounds at less than one part per million showed effective fungal growth control and no tendency to be inactivated by sludge.
Morchat [83] provided an in-depth evaluation of four commercial biocides at three different concentrations. *C. Resinae* growth was suppressed effectively by three of the biocides; whereas, only slight suppression was achieved for mixed culture fungal mat production. (Since the biocide in MIL-S-53021 was included in this study, implications of this work is deserving of detailed evaluation.)

Bennett [6] experimentally demonstrated the use of a mixture of dioxaborinanes to control microbial growth in Jet A fuel/water mixtures.

Meeks [44] provided data demonstrating that 1.4 to 5.6 ppm (w/w) of methylchloro/methylisothiazolone biocide does not degrade Jet A or diesel fuel properties and is an effective biocide in both laboratory and field tests.

**SESSION 6: METHODS FOR CHARACTERIZATION, ANALYSIS AND TESTING**

Westbrook [75] has developed a methodology for the identification of diesel fuel system debris in U.S. Army vehicles. Reference materials were collected for comparison with field samples by microscopic and other identification techniques. Sample preparation is included. The methodology has identified problem debris and, thereby, permitted diagnosis of filter plugging causes.

Sprenger [64] described a method for creating standard water-in-fuel emulsions for coalescer testing. Using a forward scattering turbidimeter to measure particle size, high shear rates across globe valves resulted in the most reproducible emulsions.

According to Treuhaft [70], the selection of fuel system components in U.S. Army ground power plants is not governed by any standards, resulting in operational problems when coupled with severe military requirements. Design guides were provided for major fuel system components. Requirements for materials, geometry, integrity, durability, and their effect on fuel quality are also covered.

Hiley [32] discussed approaches to British Navy shipboard coalescer plugging by sediment from F-76 naval distillate. Solutions include more porous coalescers, improved prefilters, and a shipboard filterability test. In this test, fuel is pumped through a fine filter at constant flow rate until a maximum pressure drop occurs or 1 liter of fuel is filtered.
Because total fuel sediment by ASTM D 2276 did not identify several cases of diesel filter plugging, Irish [35] described a Filterability Index designed to identify this problem. In this test, 1700 mL of fuel are filtered through a 5-micrometer membrane and the time required for the first and the last 100 mL is compared. A field version of the test is under development.

Solly [63] evaluated the effect of commercial additives on the filterability of mixtures of straight run with catalytically cracked stock and one automotive diesel fuel made from Australian Bass Strait crude. Sediment was produced by heating the fuel to 80°C for up to 28 days. Tertiary alkyl amine-type antioxidants decreased total sediment but gave poor correlation with filtration time. Use of dispersant alone generally led to higher particulate formation than was observed with neat fuel.

According to Reddy [58] some filter plugging of light-duty diesel engine filters was due to oxidation products, others due to microbial debris, with water increasing the problem severity. An antioxidant and a biocide should be added to fuel, and water should be excluded from fuel systems.

Halsall [86] investigated the effect of unstable diesel fuels on light-duty diesel injectors. After 2000 miles, power losses, heavy smoke, and poor starting resulted due to filter plugging by 0.1-micrometer particles. Although some nozzle coking occurred after 5000 miles, filter plugging by fuel sediment is considered the major problem.

Working with five problem jet fuels, Brinkman [11] tried to establish the reason that fuels can cause filter plugging without normal sediment formation. The work showed the importance of particle size and, therefore, filter pore size. No relationship was found between total sediment and membrane filtration time. Nylon membranes were recommended for the Filtration Time Test to avoid attack by overdoses of anti-icing additive.

SESSION 7: THEORETICAL ASPECTS OF FUEL STABILITY

Taylor [80] reviewed existing theories, particularly as regarding thermal or high-temperature stability versus storage or low-temperature stability. Major differences were pointed out, leaving the relationship between accelerated or high-temperature tests and storage stability to be essentially empirical.
Mushrush [47] studied the oxidation of sulfur compounds in benzene and tetradecane at 120°C and identified the resulting products. It proved possible to explain the oxidation product distribution in terms of a few competing reactions.

Wright [78] developed supercritical fluid analytical techniques to identify components of marine distillate sediments and compared the results to sediments resulting from thermal stress tests. Supercritical fluid chromatography coupled with mass spectroscopy offers valuable structural information regarding fuel-caused sediments.

Mayo [42] investigated soluble gum formation in jet fuels by the addition of t-Bu₂O₂ and metallic copper powder at 100°C and considered the results comparable to oxidation tests at 130°C. Several mechanisms for the resulting gum formation were proposed.

Hazlett [27] studied the role of organic acids and amine stabilizers in sediment formation with Australian marine distillates containing cracked stocks. In this investigation, the additive acted in an acid-base role rather than by free radical trapping.

Hazlett [26] added phenolic compounds from catalytically cracked cycle oil to a straight-run and a hydrotreated diesel fuel and increased deposits some 2.5 to 3 times over the neat fuel. Extracting light cycle gas oil with caustic also reduced its deposit-forming tendencies, showing the importance of phenolic compounds in deposit formation.

Hiley [31] studied naval distillate sediments which had caused filter plugging. Thin layer chromatography separated deposit components contained nitrogen and showed indole peaks. In some cases, stability additives promoted filter plugging by keeping sediment in suspension. A filterability test is being introduced into the British military fuel specification to help solve the problem.

Lee [37] categorized diesel fuel sediment by particle size as well as total weight in a program which included three fuels, three additives, and storage at 65°C, 80°C, and 95°C. A mathematical relationship between total weight and particle size was developed. The relationship between particulates and wall-adherent deposits was also studied.
SESSION 8: TEST METHOD DEVELOPMENT AND EVALUATION

Morris [9] is assessing the stability requirements of motor gasoline to assure 4-year storage life. A modified Potential Residue Test (ASTM D 873) shows promise as a control test. Engine tests will evaluate the performance of stored fuels and provide correlations with several inspection tests. Chorley provided introductory remarks (to Morris' presentation) which covered the NATO fuel requirements and pipeline/distribution system operation.

Fodor [18] is developing a test to establish the peroxide-forming tendency of jet fuel. Experimental parameters include four fuels, three oxygen pressures, and storage at 60°, 90°, and 100°C. Baseline storage is at 43°C. Peroxide-forming potential can be determined at 100°C on the basis of three of the fuels tested.

White [76] reported on a study of the variables of the Oxidation Stability of Distillate Fuel Oil (ASTM D 2274) test method. Operator technique is considered a major factor in the test's poor precision. Sediment formation rate during the normal 16 hours of the test also should be considered in applying test results.

Lee [38] described an extensive review of ten accelerated oxidation stability tests. Major variables and possible error sources include filter media, test time, and oxygen concentration. A number of changes have been incorporated into a modified D 2274, but additional changes are being considered, possibly including filtration time.

Reddy [59] stressed n-dodecane with and without six types of heteroatoms in a JFTOT (ASTM D 3241) at temperatures between 200° and 400°C. Soluble oxidation products were identified and measured. Dopants were found to affect the distribution of reaction products rather than their composition.

Kendall [84] compared the effect of fuel trace polar compounds on fuel stability in a flask oxidation test with that predicted by a JFTOT. Sulfur compounds acted as antioxidants in the flask test but increased JFTOT deposits. Acidic fractions had the opposite effect. Together they caused major increases in JFTOT deposits.
Stavinoha [85] is developing a diesel injector fouling test using the CLR-D diesel engine. Pintle deposits have been measured by a dielectric method, and air flow is now being considered as another rating technique. No relationships have been found between deposits and fuel inspection tests, but a procedure for quantitating the potential deposit-forming tendencies was presented.

SESSION 9: SHALE/COAL LIQUIDS AND ADDITIVE CHEMISTRY

Hartstein [87] in his written session opening remarks, traced the "Boom and Bust" history of U.S. oil shale from the year 1815 through the present time. While a similar story may be made for coal liquids, one fact remains inescapably the same; viz, shale and/or coal must eventually replace more of the current specification liquid and gas fuels from petroleum and natural gas stocks. The technology will eventually be developed which will allow market-oriented introduction from our diverse resources. While this session primarily dealt with stability-related considerations, the state-of-the-art in utilizing these fuels leads to the conclusion that these fuels can be compatible with today's engines and conventionally produced fuels.

Morris [46] has studied the effect of selected sulfur, nitrogen, and oxygen-bearing compounds on the stability of a shale-derived diesel fuel. None of the sulfur-bearing compounds significantly degraded the fuel; however, when tested in combinations with active nitrogen compounds, synergistic and antagonistic effects on insoluble products formation at elevated test temperatures were reported.

Beal [15] has found that the more polar extracts from unstable shale liquids exhibit the greater effect on decreasing the stability of a stable shale diesel fuel and that total fuel nitrogen content does not control the amount of sediment formed.

Through the use of Curie-point desorption mass spectrometry with factor/discriminate analysis, Hoestrey [34] found dihydroxybenzenes and hydroxynaphthalenes to be among the most reactive tar components responsible for coal-derived "retrograde" behavior. The mechanism for "retrograde" color and viscosity increases is thought to involve condensation reactions which can be prevented for several months by storing the liquids at -90°C under nitrogen.
By using compound-type separation and identification, Sutterfield [68] has been able to expand the understanding of heteroatomic hydrocarbon interactions and fates in synthetic crudes during hydroprocessing. The fate of heteroatoms (nitrogen, oxygen, sulfur, and organometallics) during the processing of coal and shale oil "synthetic" crudes is thought to be one of the most important factors to influence the future status of alternative crude sources.

Por [52] reported on studies of stabilizing shale oil, produced at the Mishor Rotem Pilot Plant, through the use of antioxidants in both neat and blends with petroleum crude oil. Further studies will look at the resultant product stability.

After evaluating the effect of nine antioxidants and four corrosion inhibitors on the JP-4 shale-derived jet fuel, Boos [7] was able to make recommendations regarding appropriate additives and concentrations for future use. Tests on the fuel test matrix included thermal stability, existent gum, particulates/filtration time, peroxides, and lubricity.

SESSION 10: EFFECT OF REFINERY PROCESSING AND ADDITIVES ON STABILITY

Henry [30] reviewed jet fuel and middle distillate additives including antioxidants, metal deactivators, and dispersants. The importance of early additive addition and consideration of solvent and chemical effects as well as stock acidity was stressed. Complex interactions can occur, and no broad additive effectiveness exists.

Weers [73] noted large differences in diesel fuel sediment formation due to differences in storage temperature. Although sediment formation was reduced by additives, this improvement had not been predicted by accelerated oxidation tests.

To overcome the formation of hydroperoxides in JP-5 fuel, Turner [71] studied the performance of currently approved antioxidants in severely hydrotreated and hydrocracked JP-5 at 60°C and 100°C. Totally hindered phenols at 8 ppm were as effective as 24 ppm of partially hindered phenols. This work is expected to lead toward revised MIL-T-5624 additive requirements.

Por [55] evaluated five oxidation inhibitors and two lubricity additives in moderately hydrotreated jet fuel in laboratory storage. Phenolic antioxidants proved effective, but adverse interactions were noted between oxidation inhibitors and lubricity improvers.
In a study of residual fuel hydrotreating, Kamiyama [36] established the effect of hydrotreating temperature and crude feed stocks on fuel oil stability. The importance of hydrotreating temperature, feed type, and solvent power of the cutter stock were demonstrated.

Smith [62] compared results from a 80°C/7-day stability test to storage at 43°C for 13 weeks using Australian diesel fuels. While it may need confirmation, the 80°C test is useful and has much better predictability than D 2274. A haze-clearing test at 200 nm is also used for additive screening.

Hardy [28] described an extensive investigation of stability additives in naval distillate fuels. Commercial amines showed the greatest improvement, hindered phenols were marginal, and phenylene-diamines were poorest.

Palmer [50] reviewed the need for color stability for Australian diesel fuel. Hydrotreating of light cycle gas oil produced a more color stable product than stabilizing additives, although the additives reduced sediment formation to an acceptable level.

Brinkman [10] presented detailed compositional analyses of acid, base, and neutral fractions of test fuels before and after testing. The color-causing species were concentrated in the acid fraction. Formation of polar oxygen-containing compounds occurred mainly in the neutral fraction which was also the fraction most responsible for sediment formation.

Posters were provided for discussion during the Conference by the authors of three papers:

- Barbee [5] showed an enhanced methodology for jet fuel clean and bright evaluation. This method was employed during a recent investigation to identify unusual "dandruff"-like particles in JP-4 being delivered to a number of Army installations. Also presented was an array of analytical data to support the conclusion that the unusual contaminant was probably delaminated epoxy-type coating material.
Malhotra [82] showed a field ionization mass spectrometric capability for advanced fuel sediment analysis and characterization. This type of analysis has expanded the understanding of the composition of some sediments from jet and diesel-type fuels.

Stavinoha [65] described a unique, nondestructive, thickness measuring device for use on ASTM D 3241 (JFTOT) test tube lacquer-type deposits. This technique employs a dielectric breakdown voltage correlation of 350 volts being equivalent to 1.0 μm of deposit thickness. The deposit-measuring device was demonstrated as an invaluable research tool for quantitating results in studies of fuel additive effects, reaction kinetics, effects of test tube metallurgy and surface finish, and the effects of fuel flow rates or residence time on deposit formation.
III. OVERALL OBSERVATIONS

- In general, jet fuel long-term storage stability appears satisfactory. Heavily hydrotreated stocks require the addition of oxidation inhibitors, primarily hindered phenols, to prevent peroxide formation.

- The storage stability of middle distillates, such as gas oils, diesel fuels, and heating oils, depends upon the amount and type of cracked stocks included. Amine-type stabilizers, as opposed to primary amine-type antioxidants, appear most successful in preventing sediment formation, but the selection of additives still seems to be pragmatic and empirical rather than based on theory.

- Although sediment weight is used by most as a criterion of fuel suitability, several investigators emphasized the lack of correlation between sediment weight and fuel filtration characteristics. The ability of particles to settle or to stay suspended as a function of particle size appears to be a major factor. Several laboratory filtration tests are under development.

- The need for a short-term test for the prediction of storage stability continues, but no test which accelerates conditions by increasing test temperature is dependable over a wide range of fuels. In this connection, several investigations using oxidation accelerators at lower temperatures appear worthy of further study.

- Microorganisms can cause storage difficulties, particularly in wet caverns. However, available biocides can control the problems, except in wet cavern storage. Some biocides were reported to be effective at low parts per million concentration in fuels.

- Several new, useful procedures are available to detect the onset of fuel corrosivity by more sensitive techniques than the standard corrosion tests.

- A new device for measuring lacquer-type deposits on thermal oxidative test tubes (ASTM D 3241) uses the principal of dielectric breakdown voltage. This method offers to be most useful in fuel thermal stability programs looking at neat and storage changes as well as additive and metallurgical effects on deposit kinetics.
New techniques utilizing mass spectroscopy and supercritical liquid chromatography are helping to understand the mechanism of sediment formation in middle distillate fuels.
IV. PROCEEDINGS OF THE "PLENARY SESSION: ORGANIZATIONAL MEETING ON THE FUTURE"

This session was opened at 4:40 p.m. on Tuesday, 29 July 1986, with the Conference Chairman presiding. A "Future Activity Questionnaire" and "Fact Sheet for Proposed Organization" (prepared by the Organizing Committee on Monday, 28 July 1986) was distributed and read to the conference attendees.

FUTURE ACTIVITY QUESTIONNAIRE

This is the Second International Conference on Fuel Stability. The organizers believe that the subject of fuel stability and handling of liquid hydrocarbons are and will continue to be of paramount importance. The organizers further believe that an association be formed that will facilitate maximum international participation in future conferences.

The attached fact sheets will help in the creation of this association. The first page is a "strawman" covering the proposed association's objectives and aims, the second sheet solicits your opinions and willingness to join and/or participate in the workings of the association.

After analyzing the questionnaires, the organizing committee will report back to the group Wednesday at 11:15 am. Therefore, please return your questionnaire as soon as possible.

FACT SHEET FOR PROPOSED ORGANIZATION

The proposed name of this international non-governmental organization is "The Association for Stabilities and Handling of Liquid Fuel Forms" referred to hereinafter as "Association".

Objectives and Aims

1. The aims and purposes of this Association are to create a platform for exchange of ideas, information, and experiences of members in the field of stabilities and handling of liquid fuel forms, including crude oils.

2. The Association shall encourage members to cooperate in research and experimentation intended to broaden the understanding of pertinent properties and processes. A major part of this function will be to sponsor periodic conferences.

3. The Association shall further, encourage, coordinate, promote and cooperate with research activities regarding stabilities and long term storage stabilities of fuels, but shall not seek to interfere with nor control policies of organizations active in this field.

Membership

Membership of the Association is open to individuals from every part of the world without discrimination of any form. An individual member may represent an organization.
QUESTIONNAIRE

1. PERSONAL INFORMATION

1.1 Name _______________________

1.2 Organization Name _______________________

1.2.1 Location _______________________

1.2.2 Function _______________________

1.3 Personal Title/Function _______________________

1.4 Is This Primarily Technical ______ Managerial ______ Administrative ______ Other (What) ______

2. ASSOCIATION

2.1 In your opinion, should a formal association for fuel stability and storage be established? Yes ______ No ______

2.2 In your opinion, should we attempt to provide coverage of areas of interest by association with an established technical organization? Yes ______ No ______

2.2.1 If Yes, which one(s) _______________________

2.3 An organization needs funds to offset costs of operation. How should such funds be obtained? by dues ______ by contributions from companies ______ by government grants ______

2.4 What dues would you be willing to pay? $10 or less ______ $15 ______ $20 ______ $25 ______ Over $25 ______

2.5 Would your employer be willing to pay your dues in such an association? ______

2.6 Would your employer, in your opinion, be willing to become an Association Member at somewhat higher dues level than paid by individual members? Yes ______ No ______

3. MEETINGS

3.1 Where should the next meeting be held (give first, second, and other choices)?

1. ________ 2. ________ 3. ________

3.2 When should the next meeting be held (what year)? 1988 ________ 1989 ________

3.3 What time of year should the meeting be held? Spring ______ Summer ______ Fall ______ Winter ______

3.4 Do you think your organization might be willing to sponsor such a meeting? Yes ______ No ______

3.5 Do you think your organization would be willing to be one of four or more sponsors? Yes ______ No ______

3.6 Would you be able to attend a meeting in

UK ______ Continental Europe ______ USA ______ Canada ______ Other ______

4. ASSISTANCE

4.1 Would you be willing to serve on a Steering Committee

a. to help plan the next meeting Yes ______ No ______
b. to develop by-laws for an association Yes ______ No ______

4.2 Would you be willing to help if you did not have to travel far to meetings of a Steering Committee? Yes ______ No ______

4.3 Would you be willing to help if you could contribute without having to travel? Yes ______ No ______
Results for the first three questions on the questionnaire indicated that (1) 10 percent of the attendees did not think that formation of a formal international association "For Stabilities and Handling of Liquid Fuel Forms" was necessary, (2) 50 percent of the attendees thought that a formal association should be formed, but under an already established technical organization with international scope, such as the World Petroleum Congress, etc., and (3) 40 percent thought that the association should be formed and chartered independent of any other technical organization.

Because of legal implications/considerations and complications voiced by some attendees with respect to joining a chartered association as representatives of their companies on a dues-paying basis, the Organizing Committee agreed to and the Chairman declared that:

(1) The International Association for Stability and Handling of Liquid Fuel Forms is hereby established as a nonchartered, nongovernmental association.

(2) The Conference Organizing Committee will now become the Association Steering Committee.

(3) The Steering Committee will form a Conference Organizing Committee when a host for the next conference is found, and

(4) Members of the new association include all attendees of the second international conference, as well as previous and future recipients of the newsletter furnished to members of the fuels industry.

The association was founded to create a platform for the exchange of ideas, information, and experience among members in the field of fuel stabilities and the handling of liquid fuels.

The nonchartered, nongovernmental, international association will encourage cooperation in research and experimentation to broaden the understanding of properties and processes in this increasingly important technical field. The association will bring together investigators in the field from all over the world to pool unclassified knowledge for the benefit of all. Periodic conferences will be sponsored by the association with the next international conference tentatively planned to be held in England in 1989.
Leo L. Stavinoha, chairman of the Association Steering Committee, will continue to prepare and distribute the biannual association newsletter. Information on the association may be obtained from Leo L. Stavinoha, Division 02, Southwest Research Institute, P.O. Drawer 28510, San Antonio, Texas 78284.
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