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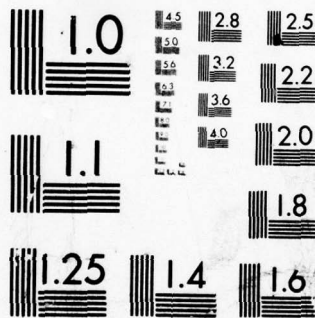
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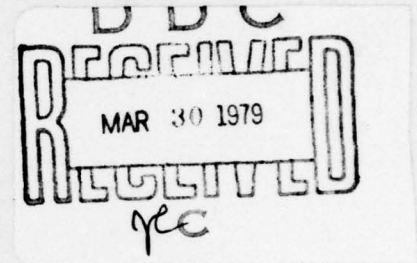
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FINAL REPORT ON CRUDE AND PRODUCT STORAGE: STATE-OF-THE-ART REVIEW AND ASSESSMENT

INTERIM REPORT
AFLRL No. 110

By
J.N. Bowden
and
L.L. Stavinoha



Prepared By

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20. ABSTRACT (cont.)

petroleum crude and product storage. Through literature review, questionnaires, and personal contacts, considerable information was identified for use in programs relating to the effect of storage on the quality of finished petroleum products. As a result of this task, it has been determined that underground storage of refined distillate products can be accomplished with proper selection of products to be stored, through specification requirements, quality control/surveillance, and judicious use of additives.

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FOREWORD

This document is the final report for Task 1 of a 4-task program, which was conducted at the U.S. Army Fuels and Lubricants Research Laboratory (USAFRLRL) at Southwest Research Institute, San Antonio, Texas under Contract DAAK70-78-C-0001, during the period June 1978 through November 1978. The work was funded through Interagency Agreement EL-78-A-01-2815 between the Department of Energy and the Department of Defense. The contract monitor was Mr. F.W. Schaekel of the Energy and Water Resources Laboratory, U.S. Army Mobility Equipment Research and Development Command, DRDME-GL, Ft. Belvoir, VA.

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The cooperation of the numerous persons who generously supplied information by answering questionnaires, responding to letters, or replying to questions at personal consultations is also acknowledged.

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I. INTRODUCTION

The Energy Policy and Conservation Act, which was signed into law on December 22, 1975, provided the legislative authorization for the creation of a Strategic Petroleum Reserve (SPR). The SPR is to provide for the storage of up to one billion barrels of petroleum in order to diminish United States vulnerability to the effects of a severe petroleum supply interruption (1)*. On May 25, 1977, SPR Plan Amendment No. 1 was transmitted to Congress as Energy Action No. 12, and became effective on June 20, 1977. The purpose of this amendment was to accelerate the development schedule to have 500 million barrels of oil in storage by the end of 1980.

A second amendment, Energy Action DOE No. 2, was submitted to Congress in April 1978 and became effective on June 13, 1978. This amendment provides for the expansion of the reserve size from 500 million barrels to 1,000 million barrels, and provides implementation for the third 250 million barrels (2).

Neither amendment considers the question of whether regional petroleum product storage should be provided for the East Coast or whether non-contiguous storage should be provided in Hawaii or Puerto Rico. A re-examination of regional and noncontiguous storage is presently underway within the Department of Energy (DOE). The results of this re-examination will be reported to the Congress at a later date.

If regional petroleum product storage is implemented, it will be imperative that the stability of the stored products be known. An investigative program was initiated to develop information to help ensure that products being considered for storage in a regional petroleum reserve would be of a quality to be immediately usable, and to identify likely

* Numbers in parenthesis refer to references in Section V.

quality assurance procedures. This program commenced through an inter-agency agreement between the U.S. Army Mobility Equipment Research and Development Command/Department of Defense and the Strategic Petroleum Reserve Office/Department of Energy. The program is being conducted at the U.S. Army Fuels and Lubricants Research Laboratory, and consists of the following four tasks:

- o Task 1 - State-of-the-Art Review and Assessment
- o Task 2 - Experimental study of the effect of storage on the quality of finished products
- o Task 3 - Update and expand data base for crude oil characteristic programs
- o Task 4 - Overall assessment and report.

This document encompasses the results of Task 1 on the "State-of-the-Art Review and Assessment" and is the final report for Task 1.

II. APPROACH

The state-of-the-art review and assessment of storage effects on crude and product quality was undertaken through: a search of the literature utilizing selected data bases; questionnaires submitted to various companies and foreign organizations having experience in long-term and underground storage of petroleum products and crude oil; and consultations with individuals knowledgeable in the area of petroleum storage stability.

A. Data Base Sources

A literature search was undertaken by computer accessing eight selected data base sources. These data bases are described as follows:

- o APILIT - Coverage dating back to 1964 of worldwide petroleum literature, including refining, petrochemicals, air and water conservation, transportation and storage, and petroleum substitutes. Prepared by Central Abstracting and Indexing Service of the American Petroleum Institute.

- o TULSA - From 1961 to date - Covers: petroleum geology, exploration geophysics, and geochemistry; well drilling, logging, completion and servicing; oil and gas production, reservoir studies and recovery methods; pollution and alternative fuels and energy sources; and petroleum transportation and storage. Prepared by the Information Services Department of the University of Tulsa.
- o ENERGYLINE - From 1971 to date - Comprehensive coverage of energy-related literature comprising over 200 core journals and selected coverage of several thousand other journals as well as reports and surveys, monographs, and newspaper articles. Subject-area coverage includes energy economics, U.S. policy and planning, research and development, resources and reserves, environmental impact, electric power transmission and storage, and fuel transport and storage. Prepared by Environmental Information Center, Inc.
- o CONDENS/CASIA - Chemical Abstracts Condensates - 1970-1971 - Bibliographic data and keyword phrases covering bio-, organic, macromolecular, applied, physical and analytical chemistry, and chemical engineering. Prepared by Chemical Abstracts Service.
- o CONDENS/CASIA - 1972-1976. Idem.
- o CONDENS/CASIA - 1977 to present. Idem.
- o NTIS - Retrospective to 1964 - A broad and cross-disciplinary file containing citations and abstracts of government-sponsored research, development and engineering reports; other government analyses prepared by federal agencies or their contractors and grantees; some reprints; and federally sponsored translations and foreign language reports. Prepared by the National Technical Information Service (NTIS) of the U.S. Department of Commerce.
- o SSIE - covers ongoing and recently completed basic and applied research in the life, physical, and social sciences. Coverage includes over 1300 funding organizations such as federal, state,

and local government nonprofit associations; colleges and universities; nonaffiliated investigators; and some non-U.S. organizations and private industry. Prepared by the Smithsonian Science Information Exchange.

A key-word hierarchy, shown in Table 1, was utilized to search the eight data bases listed above. It should be pointed out that the SSIE data base yielded no useful references. The other data bases yielded a total of 1400 citations, 300 of which were considered to be directly applicable to the stability and storage of petroleum product and crude oil. Many of these references were trade journal notices and short articles announcing various phases of the SPR program and other underground storage activities, and were not considered sufficiently technical in nature to be included in the alphabetized (Section VI) bibliography. References to underground storage of natural gas and gaseous hydrocarbons were quite numerous but also were not included in the bibliography. In addition, several environmental impact statements for various sites being considered for inclusion in the SPR program were referenced in some of these data bases, but were not used in preparing this report.

The List of References, Section V, contains the material referenced in the text of this report. These references also appear in the alphabetical bibliography where they are starred for cross referencing.

B. Questionnaires

Questionnaires of three types were prepared and sent to individuals in various companies having experience in storage of crude oil, storage of refined products, or manufacture of additives for refined products. Seven companies (including three in the United States, two in West Germany, and one each in Finland and The Netherlands) were contacted regarding their experience in the underground storage of crude oil and/or refined products. Responses were received from six of the seven. Eight companies in the U.S. were contacted regarding their experience

TABLE 1

KEYWORD HIERARCHY USED IN DATA BASE SEARCH

<u>Group A</u>	<u>Group B</u>
Petroleum	Storage
Gas oil	Containment
Gas(w)* oil	Storing
Crude	Reserve
Diesel(w) fuel	Stored
Diesel(w) fuels	Cache
Jet(w) fuel	Reservoir
Jet(w) fuels	
Fuel(w) oil	
Fuel(w) oils	
Turbine(w) fuel	
Turbine(w) fuels	
Kerosene	<u>Group C</u>
LP(w) gas	Underground
Liquid(w) petroleum	Dome
Gasoline	Cave
Middle(w) distillate	Long(w) term
Middle(w) distillates	Short(w) term
Residual(w) fuel	Tank
Residual(w) fuels	Tanks
Burner(w) oil	Subterranean
Burner(w) oils	Stability
Jet(w) A	Stable
JP(w) 4	Oxidation
JP(w) 5	Degradation
Burner(w) fuel	Strategic
Burner(w) fuels	

* (w) Words used in conjunction with each other only, and may be hyphenated.

and/or recommendations in the use of additives for increasing the storage life of refined products. Only three of the eight responded.

C. Consultations

Personal consultations were held with individuals in several companies regarding their experiences in the underground storage of refined products or crude oil. Individuals in two companies which manufacture additives were also contacted regarding their experience and recommendations in the use of additives for improving the storage stability of refined products.

III. DISCUSSION

A. History of Underground Storage

A chronological listing of events pertaining to the history of underground storage is shown in Table 2 (3). The term underground storage is generally understood to mean the storing of materials or products in unlined openings below the surface of the earth. These materials and products have been mainly of petroleum and petrochemical nature, and the underground openings may be either naturally existent or artificially created. The naturally existent openings are the gas-, oil-, or water-bearing porous spaces or fissures of sedimentary rocks or natural caverns. Storage in porous medium has found the widest application in the underground storage of natural gas and liquified petroleum gases (LPG's). These formations are either depleted gas or oil fields or water-bearing sands (aquifers). The artificially created cavities can be divided into either mechanically or solution-mined caverns. Layers of rock salt and salt domes are most suitable for solution mining as the caverns are easily created by injecting fresh water into the salt formation and removing the resulting brine. Mining by blasting and excavating can be done in nearly all types of stable rock, such as granite, limestone, sandstone, shale, anhydrite, and salt. Abandoned mines and unused tunnels may be converted into storage facilities under favorable conditions.

TABLE 2

HISTORY OF UNDERGROUND STORAGE

- 1909 The U.S. Geological Survey recommends to store the excess of natural gas underground.
- 1915 First successful underground storing of natural gas in Welland County, Ontario, Canada.
- 1916 Gas injection in the depleted Zoar field, south of Buffalo, New York, marked the first storage operation in the United States; still in operation.
- 1916 Deutsche Erdol AG (DEA) obtains a patent, covering the use of solution-mined salt cavities for the storage of crude oil and distillates.
- 1940 In Sweden first oil storage in mined rock caverns.
- 1941 Storage in combination oil and gas reservoirs was first developed by Hope Natural Gas Company in West Virginia.
- 1943 Start of LPG-underground storage (Carter Oil Co., U.S.A.).
- 1946 First aquifer storages of Muldraugh and Doe Run in Kentucky (Louisville Gas & Electric Co.).
- 1950 First solution-mined salt cavity for propane and butane put into operation at Keystonefield, Western Texas.
- 1951 More than 100 underground gas storage fields in the U.S.A.
- 1952 First large-scale aquifer storage at Herscher Dome (Natural Gas Storage Co., Illinois).
- 1954 Ethylene storage in a salt cavern starts at Fannet, Texas (Gulf Oil Co.).
- 1954 First successful gas storage in an oil reservoir (Lone Star Gas Company in its New York City field in Clay County, Texas).
- 1956 Dowell introduces the Sonar Caliper Log for the measuring of underground cavities.
- 1961 First use of salt caverns for storing natural gas in St. Clair County, Michigan (Southeastern Michigan Gas Co.).
- 1963 Old Leyden mine, Denver, Colorado, delivers stored gas for the first time.
- 1969 Abandoned mine in South Africa used for crude oil storage.
- 1969 First large-scale storage of crude oil in salt caverns starts in Europe (NWKG, Germany and Geosel, France).
- 1974 Start up of the first underground project for compressed air (NWK, Hamburg, Germany).

Source: B.O. Haudan, 1977 (3)

Depending on the type of underground storage facility, the stored products can be gaseous or liquid, and even the normally gaseous hydrocarbons can be liquified and stored under pressure (3). Although the first successful underground storage of natural gas occurred in 1915, as shown in Table 2, the storage of liquid oil products did not begin in mined rock caverns until 1940 in Sweden. Since 1966 the Federal Republic of Germany has had compulsory storage of imported crude oil and products. By January 1, 1975, a 90-day supply capacity was required of all oil companies, which included crude oil as well as fuel. Similar requirements now apply to France and other European Community countries where, in some instances, a 120-day supply is required (4). Until recently, underground storage has been used mainly for hydrocarbons. However, a new application has been found which is the underground storage of compressed air for combustion during peak-shaving at gas turbine power stations (3). The listing in Table 2 can be brought up to date with the creation of the United States SPR in 1975 and the increase of the storage goal to 1 billion barrels in 1978 (2).

B. Underground Storage of Crude Oil

In West Germany, the regulation governing minimum reserves of oil products brought about a search for economical ways of storing crude and products (4). Underground storage was known to be an economical route for accomplishing this requirement, and the creation of underground storage space in salt formations was the most economical. The geological formations in the northern German coastal region were especially suitable for solution mining of salt caverns (5). Numerous articles have appeared in the German literature on the subject of construction, maintenance, and operation of solution-mined salt caverns used for storage of crude and petroleum products (6-16). It is reported that, in West Germany, 64 salt caverns in different salt domes held 9.8 million tons of crude oil and distillates in storage at the end of 1976. An additional 13 were ready for filling at that time and 37 more were being leached or planned for solution mining. It was estimated that by the end of the seventies, the total salt dome oil storage capacity in West Germany would be increased to about 35 million cubic meters (3). A

former potassium mine in West Germany was converted for storage utilization by Winterhall AG and has a total capacity of 600,000 cubic meters of crude oil storage space (17).

The first large-scale storage of crude in salt caverns started in West Germany in 1969, but no monitoring of the condition of the stored crudes has been reported. It is estimated that some of the crude oil in the salt caverns at Lesum may have been in storage for 7 to 8 years. A project to investigate the condition of both crude and product stored at Lesum, as well as a similar facility at Etzel, has recently been undertaken by Kavernen Bau-und Betriebs-GMBH of Germany for the Department of Energy. A search of the literature revealed no systematic study of the storage stability of the crude oil, so the above investigation will be the first attempt at such a study.

In response to a questionnaire on crude storage, a representative of Mobil Oil AG in West Germany indicated that crude oils that are destined for storage are selected for low pour point and other qualities (letter from H. Rodenbusch of Mobil AG, 14 December 1977). Arabian light crudes are used extensively for storage. Mobil indicated that the maximum temperature in the salt dome caverns was estimated at 40°C. Filling of the crude oil storage caverns started 10 years ago, and it is planned to store crudes for about 20 years. Mobil does not monitor the condition of the stored crude.

In 1967, the Manosque project (85 kilometers north-northeast of Marseilles) started in France where 34 single caverns with capacities of 250 to 260 thousand cubic meters each for a total capacity of 10 million cubic meters were created by solution mining (18). Most of these caverns have been filled with crude and products. Fresh water displacement of these caverns will eventually enlarge them to 14 million cubic meters of total capacity. A storage project for 2.52 million cubic meters of petroleum in mined rock caverns was under construction in the Paris basin in 1977 at a depth of about 150 meters.

In the Scandinavian countries, it is common practice to mine storage space out of solid rock, as salt formations are nonexistent. This technique was developed during World War II for bomb shelter installations. Oil storage in rock caverns became important during the last two decades because the Scandinavian countries were completely dependent on oil imports. Finland, Norway, and Sweden all have vast installations for storage of crude oil and products in rock caverns (3).

Mined caverns were first used for hydrocarbon storage in Sweden in 1940, and the capacity of Scandinavian countries for this type of storage has been greatly expanded in recent years (3). Certain prerequisites are necessary to ensure successful storage, namely: the product must be lighter than water and relatively water insoluble; the rock must be strong enough to allow construction of fairly large caverns; and the water table must be constant and sufficiently above the level of the cavern floor to ensure a positive hydrostatic gradient. Consequently, ground water continuously seeps into the cavern where the products are stored and must be removed by continuous or periodic pumping. Thus, a bed of water exists more or less continuously beneath the stored hydrocarbons (19-22). The temperatures reported in these rock caverns are 10°-15°C. In Sweden, Arabian or Iranian light crudes are specified for storage, and Libyan crudes are sometimes used for mixing with lower pour point crudes. A pour point of -5°C is required (trip report by J.N. Bowden of USAFLRL, 23-24 November 1977.) In Finland, crudes identified as Tuymazy, Ekofisk, Agha Jari, Gach Saran, Basrah, Piper-Claymore, Arabian light, Safaniya, Kirkuk, and Hassi Messaoud have been used for storage (letter from Tuomo Saarni of Finncavern, 4 September 1978). Apparently in all the Scandinavian countries the crude oil storage, until recently, has been used for refinery supply rather than for strategic storage so the turnover time period is less than one year.

A practical method has been devised in South Africa for storing large quantities of crude oil for long periods to assure its availability during a supply interruption (23). This method is underground storage in worked out coal mines which have been converted to provide storage equivalent to a sizable oil field. A mine utilized in this manner was

in operation from June 1937 to January 1962 as a coal mine and was developed by the room-and-pillar method. After thorough study of the surrounding formations and after hydrostatic tests, conversion of the mine for storage was begun, and filling was completed in 1969 (23). Correspondence with Fenix and Scisson, Inc., of Tulsa, Oklahoma, developers of this storage program for South Africa, indicated that in 1976, crude oil had been stored in above ground tanks for 9 years and in the underground mines for 6.5 years (letter from S.E. Scisson of Fenix & Scisson, 29 November 1976). The types of crude in storage are Iranian light, Arabian light, and lesser amounts of other Mid-East crude oils. Crudes such as Iranian medium, Iranian heavy, and Cabinda have viscosities too high to be considered for long-term storage. The underground temperature in the mines is about 20°C. It has been reported that none of the crude oil stored either in surface tanks or underground has become unsuitable for use. Some evidence of emulsions at the oil-water interface has been observed as well as anaerobic bacteria growth at the same interface in both the above-ground and underground storage facilities.

In Great Britain the need for underground crude oil storage capability has been recognized, and the feasibility for this type of storage has been explored; however, apparently no underground storage is being practiced at this time (3, 24).

In the United States, the first solution-mined salt cavity was used for storage of propane and butane in 1950. A salt cavern was first used for natural gas storage in 1961 (3). In 1975 a survey of salt deposits and salt caverns and their relevance to the strategic petroleum reserve was conducted for the Federal Energy Administration (25). The feasibility of storing large quantities of crude oil in salt dome solution-mined cavities was the subject of a report to the 4th International Symposium on Salt sponsored by the North Ohio Geological Society, held in Houston in 1973 (26).

Immediately prior to and since creation of the SPR in 1975, several reports and articles have appeared in the literature on the subject of

strategic crude oil storage in solution-mined salt caverns (27-36). Initially, five sites have been obtained for SPR development:

- o Bayou Choctaw, 12 miles southwest of Baton Rouge, Louisiana with four existing cavities having a total usable volume of 36 million barrels. Allied Chemical Company has produced brine here since 1934.
- o Bryan Mound, 3 miles southwest of Freeport, Texas with 60 million barrels of usable volume in four existing cavities. Dow Chemical Company has operated brine wells here. The site could be expanded to store an additional 200 million barrels.
- o West Hackberry, 12 miles southwest of Lake Charles, Louisiana where Olin Chemical Company produces brine. Five existing cavities have a total usable volume of 57 million barrels with expansion room possible for another 160 million barrels.
- o Weeks Island, 95 miles west of New Orleans, Louisiana where Morton Salt Company has mined rock salt for nearly 75 years. Its total usable volume is 75 million barrels.
- o Sulphur Mines, 2 miles west of Sulphur, Louisiana where PPG produces brine. Three existing cavities have a total usable volume of 22 million barrels.

The eventual goal for storage of crude oil in these and possibly other sites is one billion barrels of crude oil.

Table 3 contains the purchase specification for 5 types of crude oils intended for storage in the domes at the present time.

TABLE 3

CURRENT SPR CRUDE OIL SPECIFICATIONS

Characteristic	SPR Type					Appropriate ASTM Test Method
	<u>I</u>	<u>II</u>	<u>III</u>	<u>IV</u>	<u>V</u>	
API Gravity (°API)	30-36	40-45	30-36	34-40	36-40	D 1298
Total Sulfur (wt%) max	1.99	0.25	0.50	0.25	0.50	D 1552
Pour Point (°F) max	50	50	50	50	50	D 97
Salt Content, (Lb/1000 Bbl) max	50	50	50	50	50	D 3230
Viscosity (SUS @ 60°F) max	150	150	150	150	150	D 445 & D 2161
Reid Vapor Pressure (psig @ 100°F) max	11	11	11	11	11	D 323
Mercaptans (ppm in 375°-500°F fraction) max	No limit	12	12	12	No limit	D 1323
Yields (vol%) Naphtha (375°F)	24-30	35-42	21-29	29-36	30-38	D 2802 and/or D 1160
Distillate (375°-620°F)	17-31	21-35	23-37	31-45	19-33	
Gas Oil (620°-1050°F)	26-38	20-34	28-42	20-34	23-37	
Residuum (1050°F)	10-19	4-9	7-14	0-5	7-14	
Water and Sediment (vol%) max	1.0	1.0	1.0	1.0	1.0	D 96 or D 1796

Source: H.N. Giles - SPRO - December 7, 1978

C. Effects of Storage on the Physical and Chemical Characteristics of Crude Oil

Mobil AG reported through response to the questionnaire (letter from H. Rodenbush of Mobil AG, 14 December 1977) that some of their crude oil had been in salt dome storage for 10 years; however, no inspection or testing of the crude has been conducted during this period so that any changes in the chemical or physical properties of this crude are unknown.

Reference has already been made to a project which is in progress to investigate changes to crudes stored in West Germany in salt dome cavities for 7 to 8 years.

The crude oil stored in Sweden in mined-rock caverns is generally intended for refinery use and not for long-term storage. During the building and preparing of these caverns, it was anticipated that certain problems could arise with crude oil storage (19). The caverns were constructed with provisions for heating the oil and/or the water bed so that removal of the oil from the cavern storage would be made easier. The heating is accomplished through circulation systems where the oil, the water, or, in some cases both, are pumped through heat exchangers. Two special problems were anticipated with crude oil storage: sludge and gas. Experience in Sweden has shown, however, that the sludge formation has not caused the difficulties that had been expected. Caverns are made deeper to give a water bed of 2 to 3 feet deep. The crude oil is pumped in at one end and out the other end of the cavern, and any sludge formed is allowed to settle into the water bed. Although an accumulation of sludge over long terms might cause problems, the amount of sludge after 5 to 10 years of operation has proven less than had been feared (19).

To minimize the escape of gas and avoid loss of storage volume, a number of caverns are connected together so that gas can be transferred from a cavern with a high pressure to one with low pressure. It is also possible to connect caverns to above ground storage tanks if these exist. Crude oil is stored under a pressure of 0.5 to 2.5 bar (19).

In South Africa the effect of storage on crude oil has been reported to be minimal (letter from S.E. Scisson of Fenix & Scisson, 29 November 1976). The stored crude has apparently not become unsuitable for use over the period of storage. In both aboveground and underground storage, emulsions at the oil-water interface have been observed which are difficult to break. The emulsions are believed to be caused by action of centrifugal pumps used to remove water accumulated through seepage. Growth of anaerobic bacteria at the water interface has also been observed; however, formation of H_2S was not reported. Settling of heavy ends has occurred. Mixing of different types of crude in one storage location has been avoided, especially where paraffinic and asphaltic oils are concerned.

D. Underground Storage of Products

In West Germany eight underground gas storage reservoirs were in operation in 1977 containing about 1.7 billion standard cubic meters of natural gas (3). Other projects were under construction at the time. These are referred to as porous media storage projects. In 1971 the first solution-mined salt cavern for gas storage in Germany was constructed. Since then a number of projects have been under construction for the storage of natural gas. One salt cavern in West Germany is designated for the storage of butane, and others have been constructed for ethylene and other LPG products. An old potassium mine was sealed and connected to the gas network of a nearby town to serve as a gas storage reservoir. Many of the salt domes constructed in Germany are used for the storage of gas oil under the government-mandated storage reserves program which requires that an equivalent of 90 days of consumption be kept in storage. Based on the properties indicated by Mobil AG as being representative of the materials stored in their salt dome caverns, the gas oil referred to in Germany appears to be very close to a ASTM No. 2 burner fuel.

It is reported that in France, natural gas, LPG, and distillates are stored in salt dome caverns. Propane is reported to be stored in mined rock caverns (3,18). An iron mine 200 kilometers west of Paris was

converted to five million cubic meters of storage space for fuel oil (3).

Several sources indicated that gases and other products have been stored underground in Russia for some time (3,37,38). Underground gas storage started in 1954, and this consisted of injection of gas into a depleted gas field. Aquifer storage of natural gas near large cities is extensive. In 1972 over 14 billion cubic meters of natural gas were stored. Plans exist for expansion of old storage fields and creation of new ones in aquifers and depleted zones near Moscow, Leningrad, Kiev, in the Urals, North Caucasus, Transcaucus, and West Ukraine (3). In 1964 the leaching of a large salt cavern was started, and two cavities are in operation near the Armenian capital Erevan. Haudan concluded that many LPG underground storage sites are in operation in the Soviet Union, based on reports found in the Russian literature on practical problems associated with LPG storage (3). Development and operation of underground storage sites for various products have been reported by Russian investigators (37,38,39).

The Scandinavian countries have been storing gasoline, jet, diesel, and burner fuels underground in mined rock caverns for a number of years. Personnel at the Swedish National Board of Economic Defense verbally reported that gasoline and jet fuels stored in rock caverns for 15 years have been tested and found to be in a "new" condition (trip report by J.N. Bowden of USAFLRL, 23-24 November 1977). Underground storage of fuel oil and butane in Finland and Norway has also been reported (3,40).

In response to an inquiry concerning long-term storage of products in South Africa, it was indicated that there is no experience in that country with product storage.

Underground storage of natural gas was initiated in Canada in 1915 (3). Since then, many reservoirs have been used there for storage of gas. A number of salt caverns have been in operation in Canada also for storage of gas and LPG. In Great Britain certain companies have investigated the use of old coal and anhydrite mines for gas storage but learned that

the fissures in the overlying rock and fracturing of the roof were poor conditions for this storage (3).

A report on underground storage for petroleum presented to the National Petroleum Council in 1957 (41) indicates that liquid petroleum products (liquified petroleum gases being the principal one) have been stored in a variety of underground facilities in the United States for many years. These include salt-solution cavities in salt domes, mined salt cavities, mined rock caverns, depleted oil and gas sands, water sands, stratigraphic traps, and abandoned slate quarries. Products stored in these facilities include ethylene, propane, butane-propane mixtures, isobutane, normal butane, normal isobutane mix, olefin feed stock, liquified petroleum gas, LPG blends, and natural gasoline. A report prepared by the Gas Processors Association (42) states that the underground salt dome storage capacity for light hydrocarbons in the United States was 354 million barrels in 1977. The majority of this capacity is devoted to light hydrocarbons as listed above; however, a category of "others" comprised 54.5 million barrels of capacity reported in some cases to be gasoline, condensate, and diesel fuel. Therefore, the storage of light hydrocarbons in underground salt domes and bedded salt deposits is not new to the United States; however, the storage of heavier hydrocarbons such as diesel fuels and burner fuels has not been extensively practiced. A growing number of U.S. chemical process industry companies are finding that underground facilities for storage are practical and economical (43,44).

E. The Effects of Storage on the Physical and Chemical Characteristics of Products

1. Gaseous Hydrocarbons. As stated earlier in this report, the literature was examined with respect to storage of natural gas and other gaseous hydrocarbons. Those references that were studied indicate no instability attributed to underground storage of natural gas and LPG. A need to dehydrate certain of these products on withdrawal from cavities to meet specifications was indicated in some cases. Butane, propane, and lighter hydrocarbons stored in depleted oil and gas sands and in

water sands or in stratigraphic traps generally are contaminated with residual crude oil and its dissolved gases, light gases, and water. Products so stored and contaminated may require reprocessing prior to marketing (41).

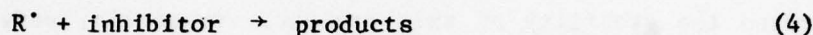
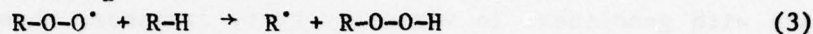
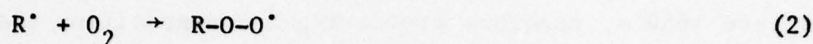
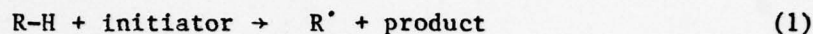
2. Gasolines. When crude oil was first refined by distillation, the various fractions, used as different types of fuels, were relatively stable, and autoxidation of these products in storage was not observed.

The demand for the fraction known as gasoline brought about the development of refinery processes, one of the first being thermal cracking. The gasoline from this process tended to oxidize and deteriorate rather rapidly in storage (45). The rearranging of the heavier hydrocarbon molecules by this process produces a considerable amount of olefins, some of which are unstable. Processes developed later such as catalytic cracking also produce a certain amount of unstable olefinic components (46). Earlier studies (45) of the composition of petroleum fuels revealed that they contain not only hydrocarbons of paraffinic, cyclic, and aromatic nature but also sulfur-, nitrogen- and oxygen-containing compounds which are deleterious to the quality of the fuel.

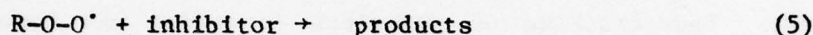
The stability of gasoline has been investigated extensively from the aspect of long-term storage and in terms of oxidation and polymerization in the tank of the vehicle to form gum. The military's interest in stockpiling gasolines prompted a number of investigations into the storage stability of these fuels (47-51). Similar studies have been conducted in Canada (52). In Russia the effect of underground storage on the properties of gasoline has been investigated (53-55). These studies have shown that, in general, the stability of gasolines varies with composition and can be improved with the use of additives. Japanese investigators have studied gum formation in catalytically cracked gasoline in storage (56).

Unstable gasoline, exposed to the action of air at ambient temperature while in storage, will undergo oxidation and some polymerization to form a resinous material referred to as gum. In the early stages of form-

ation, this material may remain in solution in the gasoline, but, due to further chemical change, will precipitate. It is generally agreed that the autoxidation of gasoline is a chain reaction involving peroxide and hydrocarbon-free radicals. The reactions that occur during autoxidation can be characterized as follows (45):



(nondeleterious)



(nondeleterious)

The second and third reactions tend to be self-perpetuating and therefore propagate the autoxidation. The inhibitors or antioxidants react with the free radicals, thus stopping the self-propagating chain reaction as shown in the fourth and fifth reactions generally resulting in nondeleterious products (45). The most effective inhibitors have been found to be phenylenediamines and hindered phenols. The chain-breaking or terminating function of these additives is thought to proceed by donation of a hydrogen atom from the reactive center of the antioxidant to the peroxy radical. The activity of the peroxy radical is sufficiently stabilized by resonance to discontinue chain propagation (57, 58). Certain metals, notably copper, dissolve, even though slightly, in gasoline and become active catalysts in the decomposition of hydroperoxides, thus supplying free radicals to the oxidation process. Metal deactivator additives will react with the dissolved copper as well as other metals to form a stable chelate, thus destroying the pro-oxidant effect of the metals (58).

Most gasoline specifications have tests to indicate the condition of gasoline at the time of the test; for example, ASTM D 381 (existent gum) and ASTM D 525-IP40 (induction period), which is also accepted as a stability predictive test. During the 1960's and early 1970's, extensive work was conducted by the Bureau of Mines in Bartlesville,

Oklahoma (now Bartlesville Energy Technology Center, Department of Energy), and by Stanford Research Institute, to investigate the nature of gasoline oxidation products generated in storage and also to develop predictive tests for storage stability of gasolines (59-61). Other tests have been developed and advanced as predictive tests with respect to the oxidative stability of gasolines (62-64).

Since the late 1950's, numerous processing and composition changes have taken place with gasolines. In virtually every instance, these changes have improved the stability of this product. Presently, very unstable thermally cracked gasoline components and polymer gasolines are almost nonexistent. Hydrocracking of petroleum fractions was begun in the middle 1940's, and the practice has increased considerably over the years. This process, reforming, and other refinery processes produce gasolines that are considerably more stable than the thermally cracked gasoline (45,57). Exhaustive hydrorefining removes not only all olefin material by saturation but also sulfur, oxygen, and nitrogen compounds. Due to these improved processes and the use of additives, long-term storage of gasolines has been practiced in Russia, Scandinavian countries, and, to a limited extent, in the U.S. with no significant deterioration of the product (53,54).

3. Jet Fuels. The storage stability of aircraft turbine fuels or jet fuels has been for some time defined and controlled through existent and accelerated gum tests. Investigations into the effect of storage on various properties have been conducted for many years in the U.S (65-73), as well as in other countries (52,74,75,76). The storage stability of jet fuel is well controlled through the extensive refining needed to meet the stringent requirements placed on jet fuels by various military and civilian specifications, and through the use of antioxidant additives (77,78). The high-temperature stability of aircraft jet fuels has been the subject of many investigations because these fuels may be subjected to thermal stresses arising during sustained supersonic flights and in some high-altitude subsonic applications (79-90). The storage stability of these high-temperature fuels has been the subject of other investigations (91-97).

Resistance to oxidation and polymerization at the operating temperatures encountered in certain jet aircraft is an important performance requirement. In high-speed flight, the fuel is subjected to considerable heat input due to kinetic heating of the airplane and also to the use of the bulk fuel as a coolant for engine oil, hydraulic, and air conditioning equipment, etc. Consequently the fuel must be able to perform at temperatures up to 250°C without formation of lacquer and deposits which can adversely affect the efficiency of the heat exchangers, metering devices, fuel filters, and injector nozzles. Research on the problem led to the development of the ASTM-CRC Fuel Coker (ASTM D 1660), which is a laboratory test apparatus for measuring the tendency of jet fuels to deposit thermal decomposition products in fuel systems. A new test for Thermal Oxidation Stability of Aviation Turbine Fuels (JFTOT), ASTM D 3241, which requires only 600 ml of sample as opposed to 6 gallons for the Fuel Coker, and takes only 2.5 hours as opposed to 5 hours, is being generally accepted for specification purposes.

Russian investigators have reported no changes in the properties (including thermal stability) of jet fuels, after 5 years of storage in rock salt caverns at 26°C, nor did the amount of sodium in the fuel exceed that found in freshly refined fuel (98). Other Russian studies show an increase in gum precursors and decrease in thermal stability after 3 years (76,99).

4. Distillate Fuels. The term "distillate fuels" is generally taken to mean fuels boiling in the range of 350° to 750°F, but is restricted to those that are used for domestic and industrial heating purposes and for diesel engine fuels. Although the boiling ranges of kerosene and jet fuels are all within these limits, they are not classified as distillate fuel oils (45). Higher boiling materials are generally classified as industrial fuel oils and called heavy fuel oils or residual fuels. Before the advent of cracking processes, and up to about 1946, distillate fuels consisted predominantly of straight run products. Although they were subject to oxidative attack, their stability on the whole was adequate for the demands placed upon them. With the increase in catalytic cracking and greater demand for diesel fuel, both thermal- and

catalytic-cracked stocks have been used in distillate fuels, which has greatly increased their susceptibility to oxidation. The oxidative attack results in the formation of soluble and insoluble materials of higher molecular weight and boiling point than the original fuel. This insoluble gum (or sediment) can cause plugging of filters, screens, and nozzles in both diesel and burner installations (45,100,101).

The storage stability of middle distillates has been the subject of numerous field storage programs in which the determination of the storage life of these fuels was the main object (102-111). Some fundamental studies led to the discovery that certain nitrogen compounds that may be present in distillates contribute significantly to sedimentation (112-114). Many of the storage stability investigations led to the development of accelerated stability and predictive tests for the storage of distillate fuels. ASTM D 2274 is perhaps the most accepted procedure for this purpose in the United States, but it is still surrounded by controversy. For this method, a measured volume of filtered fuel is aged at 95°C for 16 hours while oxygen is bubbled continuously through the sample. It should be recognized that any correlation between this test and field storage may vary significantly under different field conditions or with distillates from different sources. Work on storage stability of fuels in Great Britain led to the development of stability tests described in the British Ministry of Defense Specification DEF, Methods 16 and 17 (111). In Method 17 the sample is aged for four weeks in an oven at 49°C. It is then filtered, and the weight of the residue plus gum or lacquer remaining in the bottle is reported as milligrams of total sediment per hundred milliliters of fuel. Method 16 "ages" the fuel at 99°C for 16 hours in darkness but with sufficient air supply. These tests are reported to be of value in predicting the behavior of fuels in storage (111).

Many other methods have been proposed and are used by some companies to ensure the quality of their product (115-118). One study conducted by the Navy and reported in 1958 utilized 26 different procedures for predicting storage stability (120).

TABLE 4

GAS OIL SAMPLES FROM SALT DOME STORAGE
in
LESUM, WEST GERMANY

Cavern	Sample Date	Approximate Storage Time	Compulsory			Seasonal	
			L-104	L-104	L-105	L-106	L-106
	11/9/73	1/31/74	9/74	1/23/75	9/75	4/28/76	11/77
		Since June 1973	Since 1974			Since September 1975	
Remarks						Blanket	
Color, ASTM	0.5	0.5	0.5	0.5	0.5	2.5	L1.0
Stability, D 2274, mg/100 ml	0.5	0.4	0.5	0.8	1.3	1.3	1.0
Neutr. No., mg KOH/g	0.06	0.08	0.06	0.06	0.03	0.06	0.11
Spec. grav., g/ml @ 20°C	0.833	0.834	0.833	0.833	0.834	0.834	0.836
Sulfur, wt%	0.28	0.25	---	0.39	---	---	---
Cloud Point, °C	-8	-7	-7	-7	-4	-7	-6
Distillation, °C							
10% recovered	200	197	205	200	213	204	210
50% recovered	---	249	258	255	265	256	264
95% recovered	343	335	342	343	---	---	346
85% recovered	---	---	---	---	323	314	318
Flash Point, °C	60	---	73	67	74	53	75
Sodium, ppm	---	---	---	---	0.26	0.12	---

Testing in Progress

Russian investigators have reported that two similar diesel fuels that differed in naphthene content and acidity were stored for over 40 months in contact with brine. Analyses after the storage period showed an increase in acidity with no change in other properties (121). Other Russian investigators reported no effect under similar conditions (122).

Special refinery treatment of thermal- and catalytic-cracked stocks has been found to improve distillate fuel stability (123,124) and use of antioxidant, metal deactivator, and dispersant additives also improves stability (125-128) (letter from C.P. Henry of E.I. duPont de Nemours & Company, 8 September 1978; letter from J.D. Bartleson of Ethyl Corp., 23 August 1978). One company especially interested in long-term fuel storage intended for use in emergency standby generating equipment recommends the use of a dispersant containing a metal deactivator (but not anti-oxidants) as the most effective stabilizing additive package for diesel fuels. Fuel shelf life up to 20 years is claimed (129) (letter from G.H. Kitchen of Bell Labs, 2 March 1978). This experience was also addressed in a recent report on switching of fuel oils during a national emergency (130). Federal Specification VV-F-800B, Fuel Oil, Diesel, and MIL-F-16884-F, Fuel Oil, Diesel, Marine, contain lists of approved anti-oxidants for use in these fuels, and the marine diesel fuel specification also contains a list of approved metal deactivators.

Reviews of stability and deterioration of distillate fuels were published in 1966 (131) and 1977 (132). Mobil AG has monitored gas oil stored in salt dome caverns in West Germany since 1973, and the results are shown in Table 4 (trip report by J.N. Bowden of USAFLRL, 23-34 November 1977). The materials appear to have the same boiling range as a No. 2 distillate fuel, and no deterioration of the fuel is apparent based on these data (D2274 values, sodium levels and lack of particulates). A German firm under contract to DOE is currently performing detailed analyses on samples of the gas oil from one of the Lesum salt dome caverns.

If a water-fuel interface exists when the fuel is in storage, bacterial and fungal activity may occur. This activity has been the subject of

many investigations involving surface storage, and it is generally agreed that it can best be arrested by good housekeeping practices, such as ensuring a minimum water bottom in the storage container (133-138). Certain biocidal additives have been found to be effective in controlling microbiological activity (139-141). Storage of distillate fuels over brine may tend to reduce microbiological growth (trip report by L.L. Stavinoha of USAFLRL, 21 July 1978). Biological activity has not been observed in the salt dome storage projects; however, anaerobic bacterial activity was suspected in the mine storage of crude oil in South Africa (letter from S.E. Scisson of Fenix & Scisson, 24 November 1976).

5. Heavy or Residual Fuel Oils. Instability in residual fuel oils may manifest itself either as waxy sludge deposited at the bottom and sides of an unheated storage tank or as fouling of preheaters while heating the fuel to elevated temperatures. There is much speculation regarding the mechanism of sludge deposition. It may be due to oxidation, polymerization, or the method of producing the fuel. Whichever process is involved, insoluble compounds are formed which eventually settle to the tank bottom and form sludge (142). Asphaltene deposition may result from the mixing of fuels of different origin and treatment. Straight run fuels from the same crude source are normally stable and mutually compatible. Heavy fuels produced from thermal cracking and visbreaking operations can be incompatible if blended with straight run fuels. Blending of No. 2 fuels with residual fuel from cracking or visbreaking processes may result in asphaltene precipitation (143). The general mechanism of sediment formation in distillates has been reviewed (45) and sedimentation rates calculated for No. 4 vs. No. 6 fuel oils (letter from B.L. Mickel of Amoco Oil Co., 17 August 1978).

Light residual oils may contain waxy components which will separate if the fuel is stored at cool temperatures for extensive periods. The presence of waxy materials can have ill effects on handling of the fuels, thus cloud and pour point limits are generally specified when purchasing these fuels. It has been found, however, that these tests fail to predict the pumpability temperature of the fuel (143) after some

period of storage, and pumpability testing has been a major concern of the industry and the British Admiralty since the early fifties (144). As a result, a number of investigations into this problem were conducted, and several technical reports were published in the Journal of the Institute of Petroleum, culminating in the development of a pumpability method designated IP 230/69 (145-154). ASTM has also developed a method, ASTM D 3245, designed to give the minimum cold storage and minimum handling temperatures which may be used for a given fuel oil.

In 1960 a construction company in Sweden began building underground storage caverns in rock formations for oil companies and for the governments of Sweden, Finland, France, and other European countries (letter from S. Bylund of SENTAB/Svenska, 25 September 1978). Various designs are used for these caverns, depending on the material to be stored and whether or not the facility is for refinery storage. If the storage is for crude oil or heavy fuel oil and especially if it is for reserve storage, then a system for heating the oil, the water bed, or both is designed into the construction of the storage cavern. To facilitate withdrawal of the oil, the storage temperature in these types of caverns is maintained at 50° to 60°C.

A hydrotreated-vacuum oil similar to ASTM No. 4 burner fuel has been proposed as a candidate for the regional product storage program under consideration by DOE. Table 5 compares the suggested properties of this product to the requirements for marine diesel fuel, a navy distillate fuel, and ASTM No. 4 burner fuel. In the middle 1960's the U.S. Navy began converting selected steam-boiler ships to the use of a distillate fuel described by Military Specification MIL-F-24397, Navy Distillate (ND) (108). This fuel (the use of which has since been discontinued) was a higher boiling material than diesel fuel marine (DFM) and approached the viscosity range of a No. 4 fuel. A program was conducted to evaluate the storage characteristics of four fuels meeting the requirements of the ND fuel.

The fuels were stored in columns, bottles, and cans for 3 years. This period was considered indicative of field storage experience. The

TABLE 5

PROPERTIES OF VARIOUS FUELS

	<u>SPRO Proposed Fuel*</u>	<u>Diesel Fuel Marine(DFM)</u>	<u>Navy Distillate</u>	<u>ASTM No. 4 Burner Fuel</u>
Distillation, D 86, °F				
10% Distilled, max	---	---	500	---
50% Distilled, max	---	---	644	---
90% Distilled, max	---	675	740	---
95% Distilled, max	---	---	765	---
End Point, max	---	725	---	---
Residue plus loss, % max	---	3.0	---	---
Viscosity, SUS at 100°F	45-125	31-41	58.9 max	45-125
Flash Point, °F, min	130	140	150	130
Pour Point, °F, max	20	20	25	20
Ash, wt%, max	0.10	0.005	0.01	0.10
Water and Sediment, v %, max	0.50	---	0.02	0.50
Sulfur, wt%, max	0.30	1.00	1.30	legal
Carbon Residue, Ramsbottom, wt%, max	0.3	0.2	0.4	---
Compatibility with a standard fuel oil	yes	---	---	---

* Possible additional requirements are:

20 v% max of cracked stocks

no residual stocks

10 to 20 pound/thousand barrels (PTB) amine type oxidation inhibitor

0.5 total PTB metal deactivator

0.5 total PTB corrosion inhibitor

a dispersant additive

results of these storage experiments were correlated with accelerated experiments using ASTM D 2274 and a 110°F oven storage procedure. It was concluded from this program that the ASTM D 2274 procedure was probably a suitable method for predicting the storage characteristics of the navy distillate fuel.

IV. SUMMARY

The practice of storing petroleum products and crude underground began in 1915 with the successful storing of natural gas in old gas wells in Welland County, Ontario, Canada. Since then, two types of underground storage have been used in various parts of the world. These are porous media and cavities. The porous media include depleted gas or oil fields and waterbearing sands, also referred to as aquifers, and are utilized primarily for storage of gaseous materials such as natural gas, butane, propane, butylene, ethylene, propylene, and air. The cavities may be abandoned mines, mined cavities in granite, rock salt or other suitable rock formations and in solution-mined cavities in salt. Liquid products, including LPG, gasoline, distillates and crude oil, have been stored in salt cavities and in mined cavities, and abandoned mines have been used for crude oil and distillate storage.

Until recently, long-term, strategic storage of crude oil has taken place only in a few instances in Europe and in South Africa. Based on what is known for this limited number of cases, it appears that the refinability of crude oil will not be affected by prolonged storage. Most crudes will likely deposit a "sludge" during storage which may interfere with withdrawal pumping. It is speculated that this sludge is composed of microcrystalline wax, sand, water, and possibly asphaltenes. Emulsions at the water-oil interface have been reported after prolonged storage and these have been attributed to the action of centrifugal pumps used to remove accumulated seepage water. It is possible that these emulsions resulted from biological activity, although the only such activity reported was thought to be anaerobic (a process that generally produces hydrogen sulfide), but no detection of H₂S was noted. The bitumen sediment and water inherently present in crude may contribute to this layer by settling out during quiescent storage.

Underground storage of products, including natural gas, LPG, gasoline, diesel fuel, burner fuel, gas oil, and jet fuel has become common practice in Europe. The lighter products, natural gas through gasoline, and, in a few instances, diesel fuel have been stored in salt cavities and mines in this country for varying periods of time since about 1943. There are no reports of deterioration, although these are mostly short-term storage programs (one year or less) as opposed to long-term reserve storage programs.

Several governments, notably West Germany, France, and other European Community countries, have imposed regulations that reserves of products must be kept by various oil companies to ensure against petroleum shortages. Salt dome cavities and mined rock caverns are typically utilized for this product storage, and even though storage temperatures range from a low of approximately 5°C in mined rock caverns to a high of 50°C in some of the salt dome caverns, none of the stored products was reported to be unstable even after a number of years. One instance of a relatively high sodium content in a gas oil (equivalent to a No. 2 fuel oil) stored in West Germany was observed. However, by blending with a fresh refinery product at a 2 to 1 ratio, this fuel was utilized with no further problem.

In general, the reports reviewed and the personal contacts made indicate that long-term underground storage of refined distillate products has been accomplished with proper selection of products to be stored, through specification requirements, quality control/surveillance, and judicious use of additives to control any potential oxidation, sediment formation, biological activity, etc.

No experience with extended storage of heavy or residual fuels has been reported in the literature, although correspondence related to this study indicates the Scandinavian countries have had experience in this area. The main concern with storage of this fuel is wax and sludge separation, which is controlled by continuous dynamic heating of the fuel.

A program has been initiated in West Germany, under a contract between KBB GmbH and the Department of Energy, to sample and analyze crude oil and product stored in two different salt domes. Results of this effort are not yet available but will be informative when completed.

While considerable information was gained on the prolonged storage of crude oils and products, no specific guidelines were identified for purchase specifications, quality control/surveillance, and for handling of jet or heavy burner fuels. Considerable information was identified for use in programs relating to the effect of storage on the quality of these finished products.

V. LIST OF REFERENCES

(These same references are also contained in the Section VI, Alphabetical Bibliography.)

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