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Part III

STORAGE STABILITY OF HIGH TEMPERATURE FUELS

M. L. Whisman and C. C. Ward

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FOREWORD

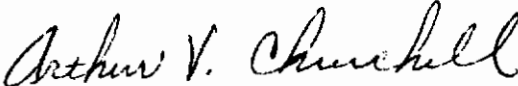
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This report covers work conducted from February 1966 to February 1967, the third year's effort of a three-year contract.

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This technical report has been reviewed and is approved.


Arthur V. Churchill, Chief
Fuels, Lubrication & Hazards Branch
Support Technology Division
AF Aero-Propulsion Laboratory

ABSTRACT

This report covers an investigation of the influence of fuel constituents during storage on thermal stability of Spec MIL-J-25656B type (JP-6) and other high temperature fuels. The major objectives of the program were satisfied by the successful development and evaluation of several methods utilizing radio-tracers to determine the causes of thermal instability as well as predicting thermal stability quality of high temperature fuels during storage.

Ninety-four test blends of 12 fuels and 9 radioactive fuel compounds were prepared and tested in the evaluation of a microscale thermal stability procedure developed for the purpose of determining the contribution of selected fuel components to thermally induced deposits. Eighty-eight of these test blends were retested after 26 weeks storage at 130° F to determine the effect of storage upon deposit-forming tendencies. Analysis of the data obtained in this study resulted in a further study of thermal decomposition of a fuel antioxidant additive.

An investigation of several variables associated with an in situ hydro-generation method using tritium as the index of reactivity was completed as an effort to develop a sensitive and accurate analytical technique for very low concentrations of olefins in hydrocarbon fuels.

In addition, initial steps were taken toward obtaining or synthesizing radioactive materials and reference standards essential to a continuation of this program along a similar line of approach.

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I. INTRODUCTION

This report reviews the experimental work conducted by the Bureau of Mines during the 12-month period from February 1966 to February 1967, representing the third year's work under a 3-year contract with the Air Force. Details of the major objectives of this contract are to be found in a previous report (1) and can be summarized briefly as follows:

1. Develop test methods utilizing radiotracers which will have better sensitivity and precision than present analytical techniques for measuring the degree of participation of fuel components in deposit-forming reactions.
2. Based upon the most promising of several radiotracer approaches, further development will be concentrated on one or two test methods and applied to a selected number and variety of high temperature fuels.
3. Resultant data will be evaluated in an effort to obtain a better understanding of gum-forming and degradation processes associated with fuel storage and thermal stresses.

Experimental work during the first and second year concentrated on objectives 1 and 2 listed above. Test methods that were developed and evaluated included one technique for measurement of radioactive deposits formed in a 5-ml static bomb following thermal stressing and three chromatographic procedures for separation of existent gum from a fuel matrix. Test fuels and special blends used in this program are listed in Appendix I.

II. MICROSCALE THERMAL STABILITY DEPOSITS

A. Background

A test procedure employing modifications of a microscale thermal stability test procedure is described in Appendix II of this report. The procedure was developed to show contribution of selected fuel components to deposits that were formed upon heating a high temperature jet fuel. The method was developed so that deposits adhering to the walls of the microbomb, as well as filterable deposits, could be measured with excellent sensitivity. Radioactivity associated with adherent deposits was measured by converting the small bomb to a proportional counting device, and filterable deposits were removed from the fuel by means of cellulose ester filters and measured by liquid scintillation techniques. A total of 94 blends representing the 12 test fuels of this program and 9 carbon-14 labeled radiotracers were prepared, tested, and stored at 130° F. Sixty-four of these blends were completely tested and reported previously (2).

B. Application of Test Procedures

Twenty-four blends of the 12 test fuels of this program and 2 carbon-14 labeled radiotracers were prepared, tested, and stored at 130° F. Storage and analysis of these samples completed this phase of the test program including 88 blends of which 64 were previously reported. Each blend was stored for 26 weeks in an amber glass bottle in a laboratory oven and retested by the established procedure.

Tables 7 through 18 relate to 12 blends, each containing 3 ppm of indan-2-C¹⁴. This radiotracer made little contribution to adherent bomb deposits before storage, and only 2 of the 12 blends showed a significant increase in adherent bomb deposits after the storage period. However, after heating the blend, 7 of the 12 blends showed an appreciable amount of radioactivity in filterable material and nearly all of the 12 test blends showed significant increases of indan contribution to filterable deposits after the storage period. A graphic presentation of the contribution of indan to total deposits after storage can be seen in figure 1. The percent of radiotracer in total deposits (adherent plus filterable) is plotted versus the test temperature. The adherent deposits were included in this figure because they were very low compared to filterable deposits and inclusion of the adherent values did not appreciably change the shape of these curves. It should be pointed out that these curves were generated from only three points plus the room temperature control value, and therefore detailed interpretation and extrapolation of these data is not possible. However, it is worthy of note that these curves are of the same general shape and the magnitude of reaction is similar to data obtained with other aromatic type radiotracers and reported in previous reports (1) (2).

Tables 19 through 30 were prepared from data obtained from 12 blends, each containing about 2.5 ppm of carbon-14 labeled n-hendecane (an 11-carbon atom normal paraffin). These blends showed no contribution of the radiotracer to adherent bomb deposits before or after the 26-week storage period. Although this paraffinic radiotracer did not contribute appreciably to filterable deposits before storage, there was a significant and surprising increase of contribution following the 26-week interval at 130° F. The average increase was about fourfold as compared with only a twofold to threefold increase in similar blends containing a radioactive olefin as the radiotracer. Figure 2 is a graphic presentation of the contribution by hendecane to total deposits after the accelerated aging (26 weeks at 130° F). This family of curves is closely grouped and the implication is that the extent of hendecane reaction is independent of the test fuel matrix of these blends.

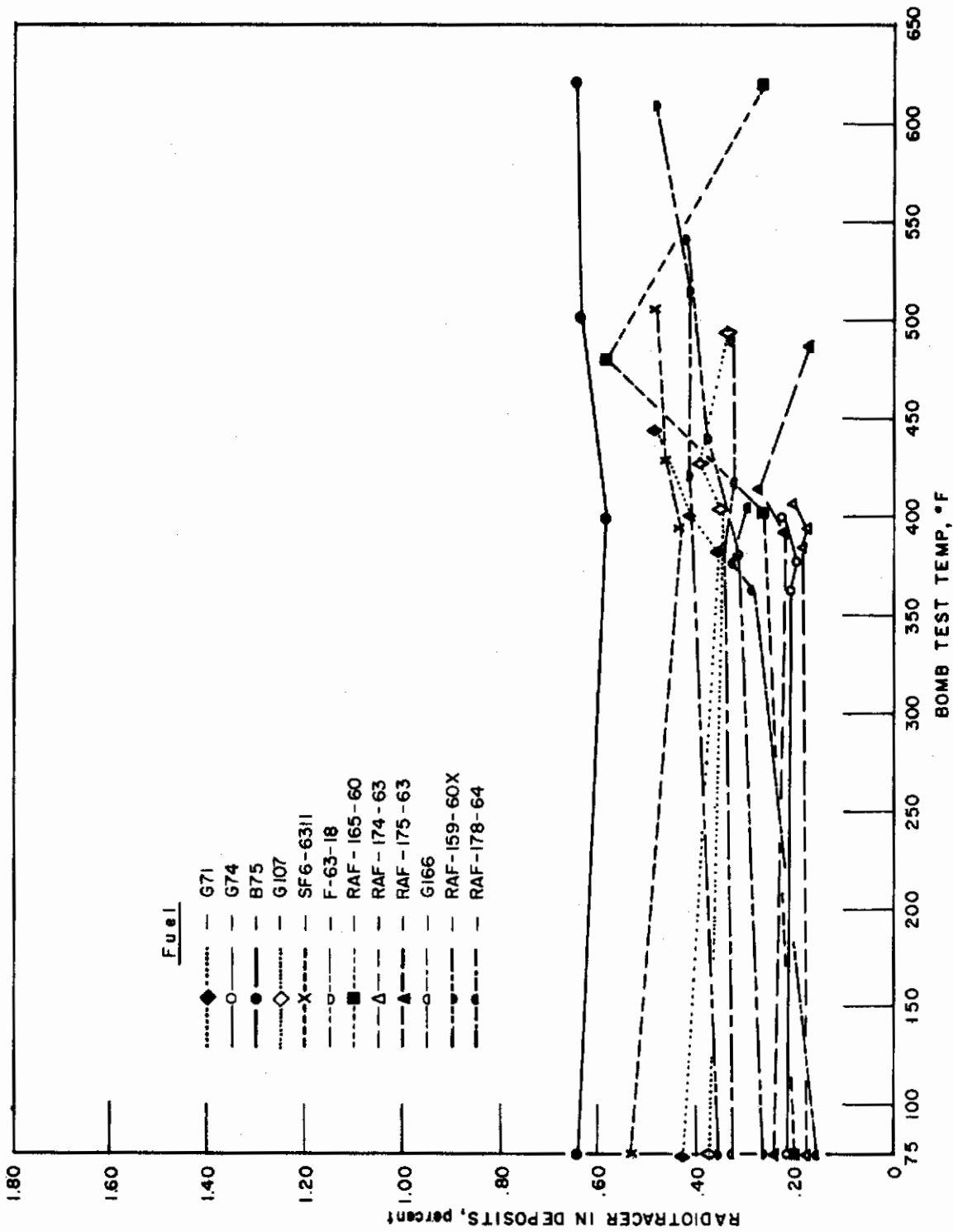


FIGURE 1. - Contribution of Indan-C^{1.4} to Total Deposits Formed During Thermal Stability Evaluation of Twelve Test Blends Stored 26 Weeks at 130° F.

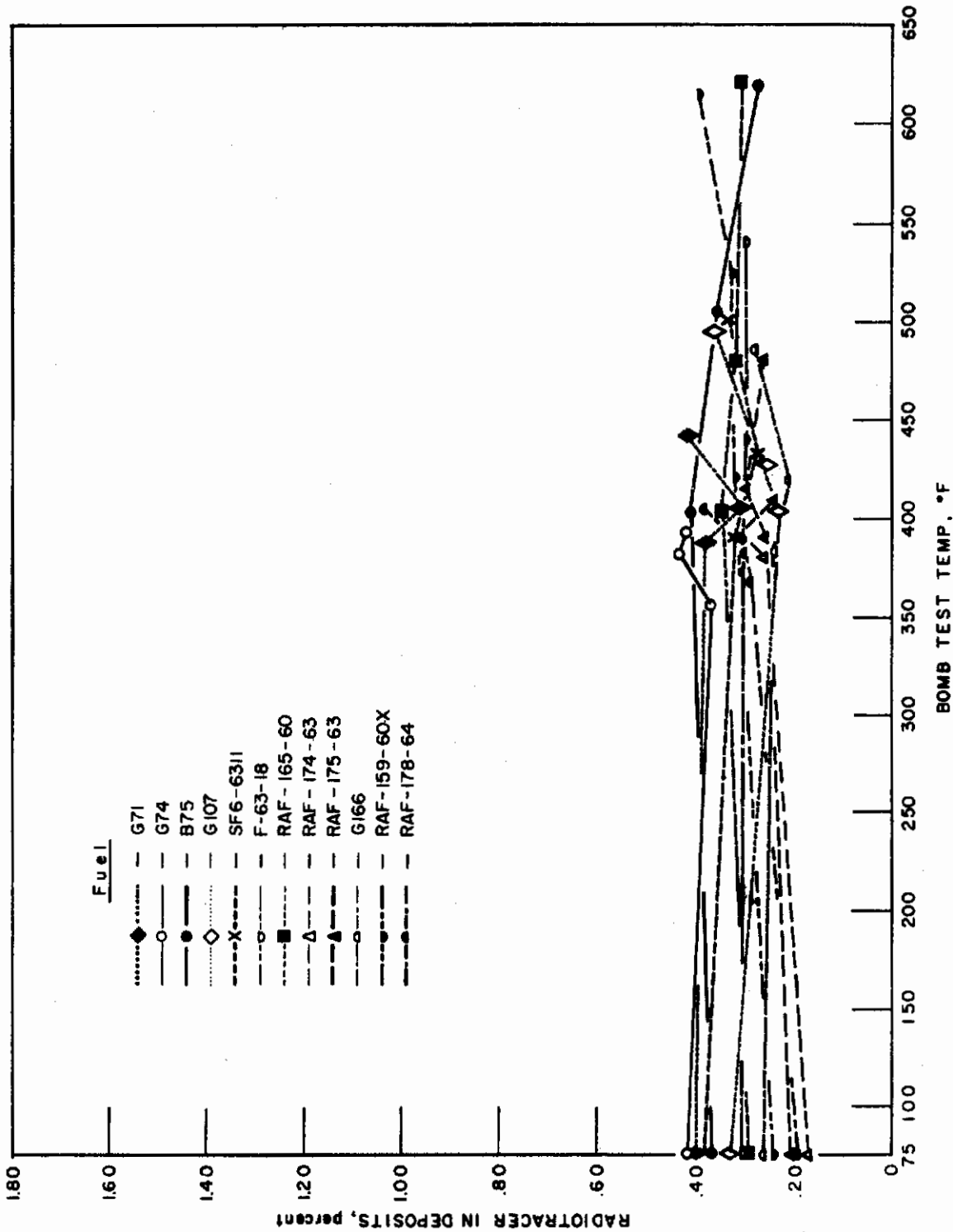


FIGURE 2. - Contribution of n-Hendecane-C¹⁴ to Total Deposits Formed During Thermal Stability Evaluation of Twelve Test Blends Stored 26 Weeks at 130° F.

Tables 31 through 36 were compiled from data obtained in making radioactive material balances for some of the above test runs. The total radioactivity in 5 ml of the blend (quantity required for one test) should just equal the total radioactivity recovered as adherent deposits, filterable deposits, and in the filtrate. At room temperature and only slightly elevated temperatures, the radioactive material balance was good. However, appreciable losses of radioactivity occurred above 400° F in some blends that were discussed in a previous report. Tables 31 and 32 show this radioactivity balance for two of the test blends containing indan. The imbalance shown in table 31 at room temperature probably resulted from light-end fuel evaporation during the vacuum filtration rather than from a chemical change of the radiotracer. However, an appreciable loss was noted for this blend at elevated temperatures. The losses associated with blend -900 shown in table 32 are probably all associated with vacuum filtration procedures. The data in tables 33 through 36 associated with material balances of n-hendecane indicate no significant loss of radioactivity.

C. A Summary of Test Data

A comparison of all data obtained by microscale thermal stability testing of the 88 test blends of this program was accomplished by means of bar graphs that show the extent of contribution of 8 radiotracers to thermally induced deposits in each of the 12 test fuels.

Data from table 37 are summarized in figure 3, indicating the extent of reaction of hendecane as measured by total deposits at 400° F in each of 12 test fuels. It was anticipated that the contribution of normal paraffins to deposits would be negligible, and this is borne out by the results obtained on the fuels before storage. Results on the stored fuels indicate that the reactivity of the hendecane is affected adversely by storage in a fuel environment for 26 weeks at 130° F. Although the increase of radiotracer contribution after storage is surprisingly large, a significant variation between test fuels is not evident. This relatively uniform increase of radioactivity in thermally induced deposits after storage might be explained by peroxidation of the radiotracer. The peroxide would be destroyed by subsequent heating of the blend resulting in fragmentation of the labeled molecule. Recombination could then lead to the results observed in this series of test blends.

Data from table 37 are shown in figure 4 which is a similar presentation for an olefinic radiotracer. Initial testing before storage disclosed a fairly uniform and low level of reaction with the exception of the blend containing fuel -178-64, which has the lowest thermal stability of the 12 test fuels. However, this same blend showed no sensitivity to storage in terms of increase of olefin participation in deposit formation. The largest increase of radioactivity in thermally induced deposits from olefin blends after storage was observed in

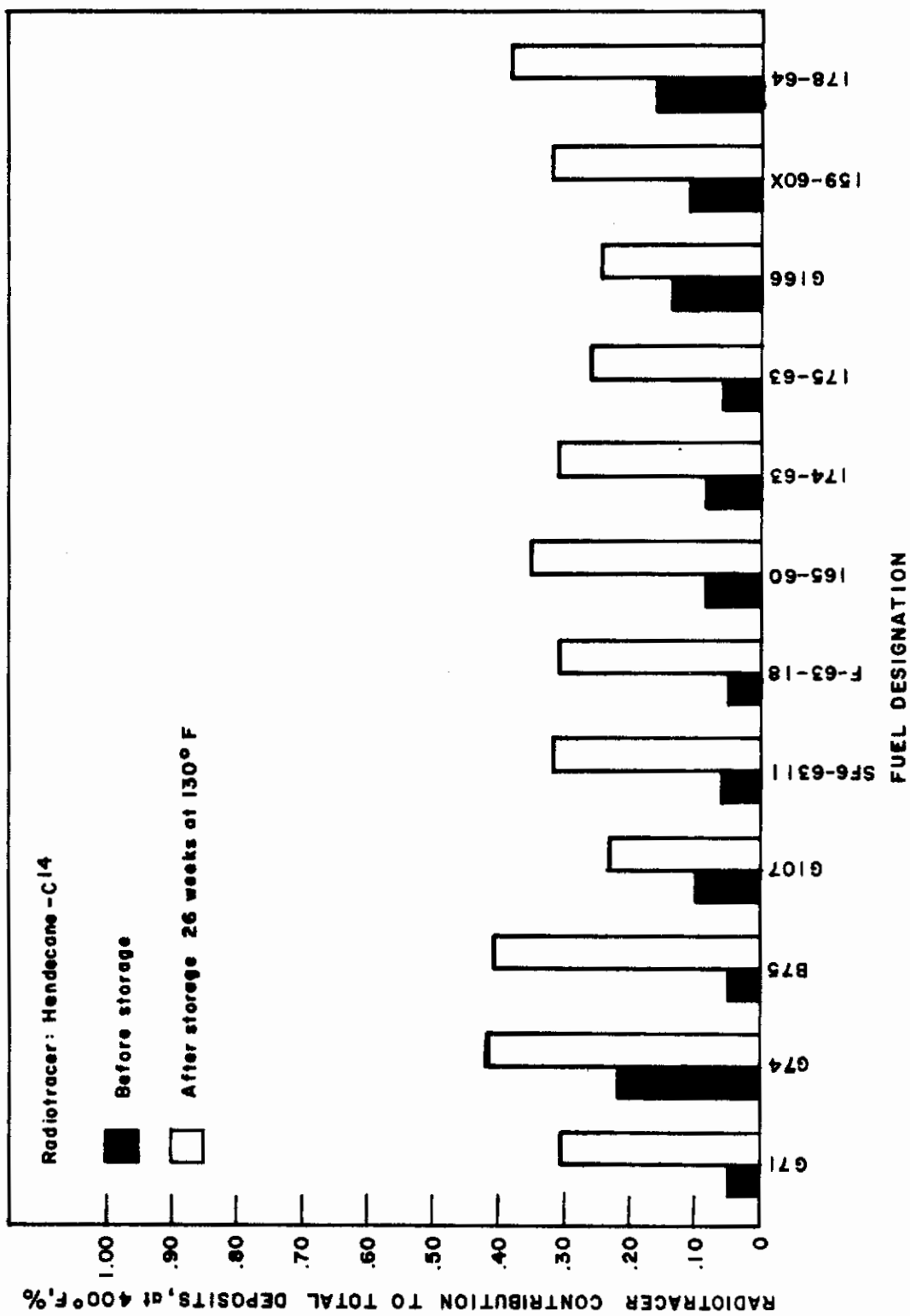


FIGURE 3. - Contribution of Hendecane-C¹⁴ to Thermally Induced Deposits at 400° F.

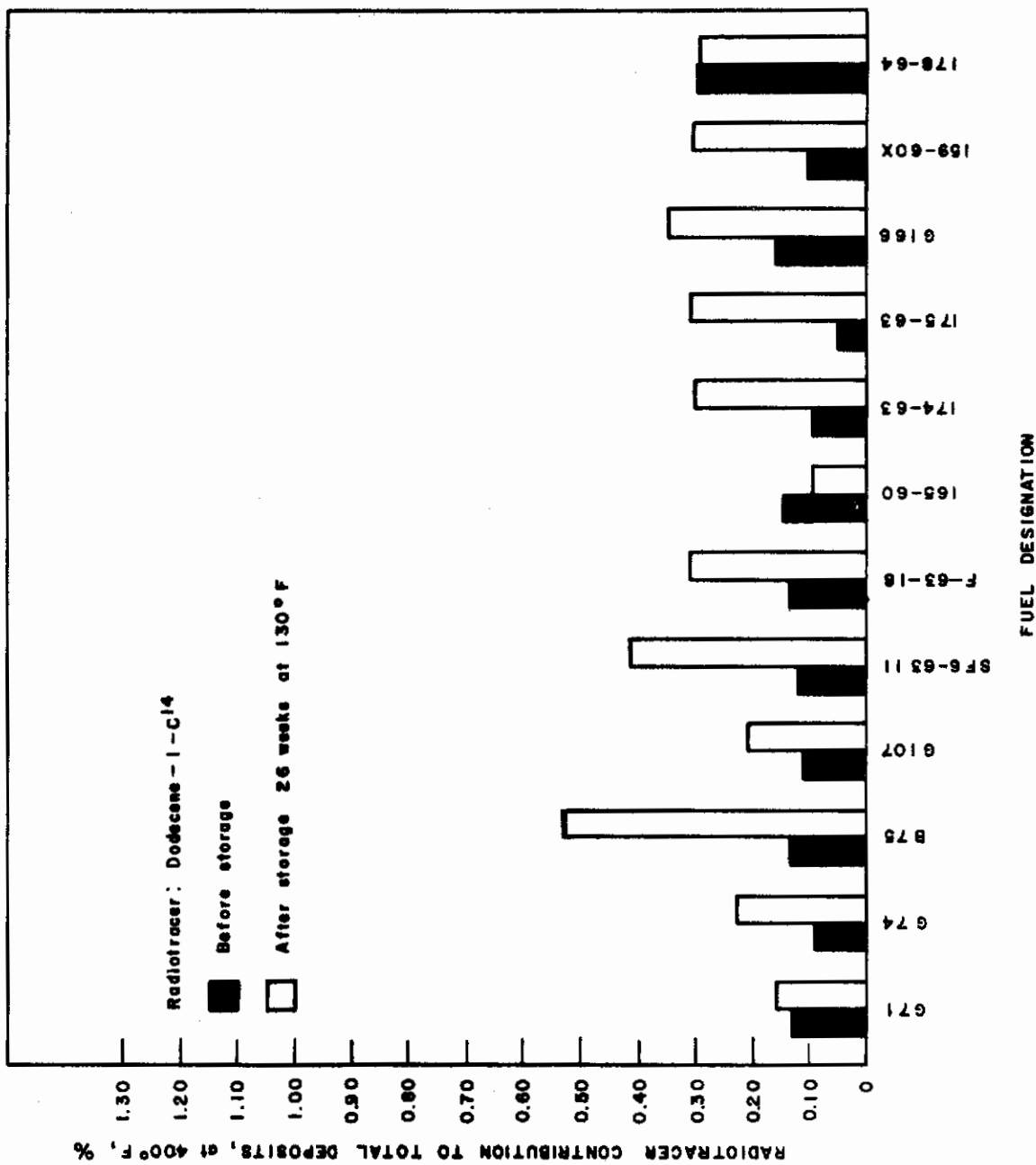


FIGURE 4. - Contribution of Dodecene-1-C¹⁴ to Thermally Induced Deposits at 400° F.

a fuel matrix of -B75 which has a reported ASTM-CRC coker failure temperature of 500° F, indicating good thermal stability. This fuel has 2.4 percent olefins by FIA analysis, which approaches the highest olefinic content of any of the 12 test fuels. It would seem unlikely, in this light, that an additional 2 ppm of radioactive olefin would significantly affect the deposit-forming tendencies of the fuel. This conclusion is borne out in data to be subsequently discussed that shows the storage of this particular fuel blended with a variety of fuel components increases the deposit-forming tendencies.

Figure 5 is a graphic representation of data from table 37 obtained from 12 blends, each containing about 3 ppm of a radioactive tetramethylbenzene. The highest initial contribution of this radiotracer to thermally induced deposits was observed in blends containing fuels -G74 and -178-64. Both of these fuels are of low thermal stability as defined by the ASTM-CRC coker. However, following the accelerated aging period of 26 weeks at 130° F, these same two blends showed the least change while some of the higher quality fuels showed large increases, percentagewise, of radiotracer reaction. The largest percentage increase was observed in the blend containing fuel -175-63. This fuel has 12.9 percent of total aromatics as determined by FIA analysis. Since this is a relatively high amount of aromatics for supersonic aircraft fuels, it seems unlikely that the additional radioactive aromatic was instrumental in degradation of thermal stability quality of this fuel.

Similar data from table 37 for another aromatic, diphenyl, generally considered to be quite thermally stable in itself, are shown in figure 6. Only five test blends with this radiotracer were prepared and analyzed. The results from three of the five blends indicated that storage had no appreciable effect upon the degree of radiotracer participation in deposit-forming reactions.

Deposit-forming tendencies of another aromatic, indan, are graphically displayed in figure 7. At least half of these test blends showed high initial contribution of this radiotracer to deposits as determined by the microscale thermal stability test. The percentage increase of deposits in each blend after storage was less dramatic than in some of the other blends. Tetralin, for example, shown in figure 8, is a compound similar to indan but is known for its tendency to form peroxides under favorable circumstances. These data, taken from table 37, showed very significant increases of tetralin participation in thermally induced deposits following the accelerated aging period. Especially dramatic were the increases observed in three blends representing fuels -B75, SF6-6311, and -G166. These three fuels contain 1.3 percent, 11.2 percent, and 12.9 percent aromatics respectively and have ASTM-CRC coker failure temperatures of 500, 450, and 425° F. Neither of these fuel properties appear related to the high reaction extent of tetralin. However, the highest percent of indan contribution to deposits was also observed in fuel -B75 and it becomes increasingly

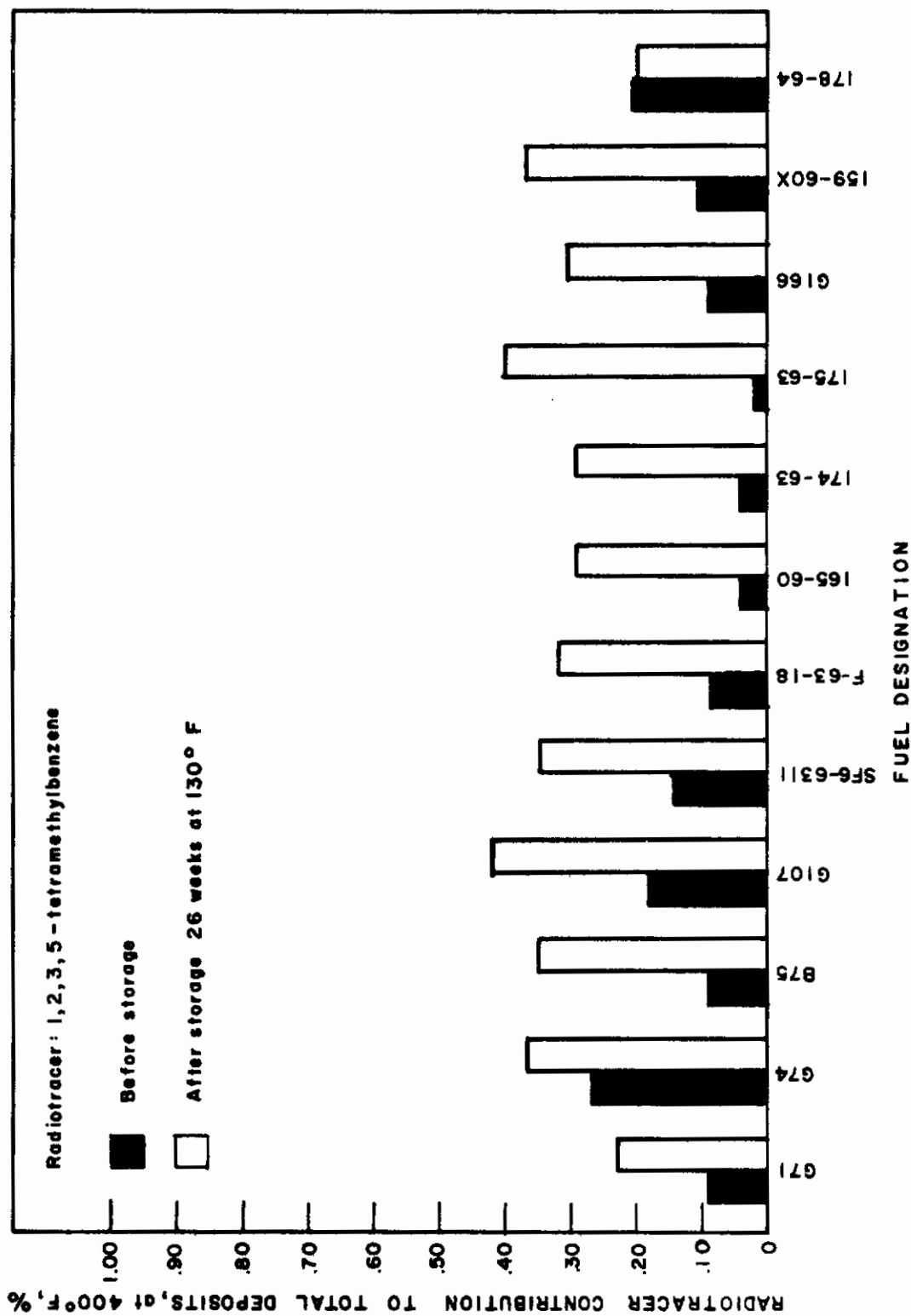


FIGURE 5. - Contribution of 1,2,3,5-Tetramethylbenzene-C¹⁴ to Thermally Induced Deposits at 400° F.

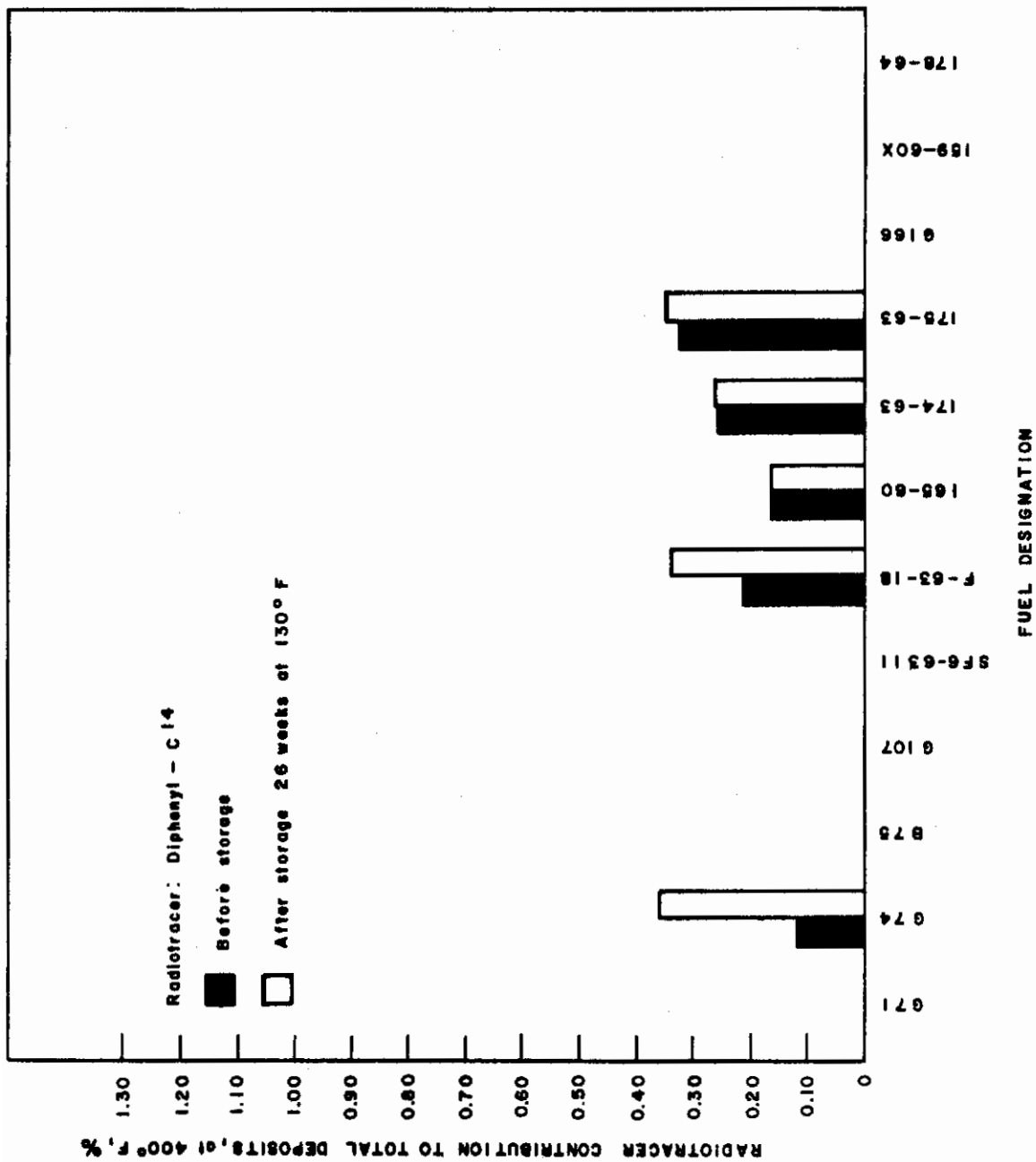


FIGURE 6. - Contribution of Diphenyl-C¹⁴ to Thermally Induced Deposits at 400° F.

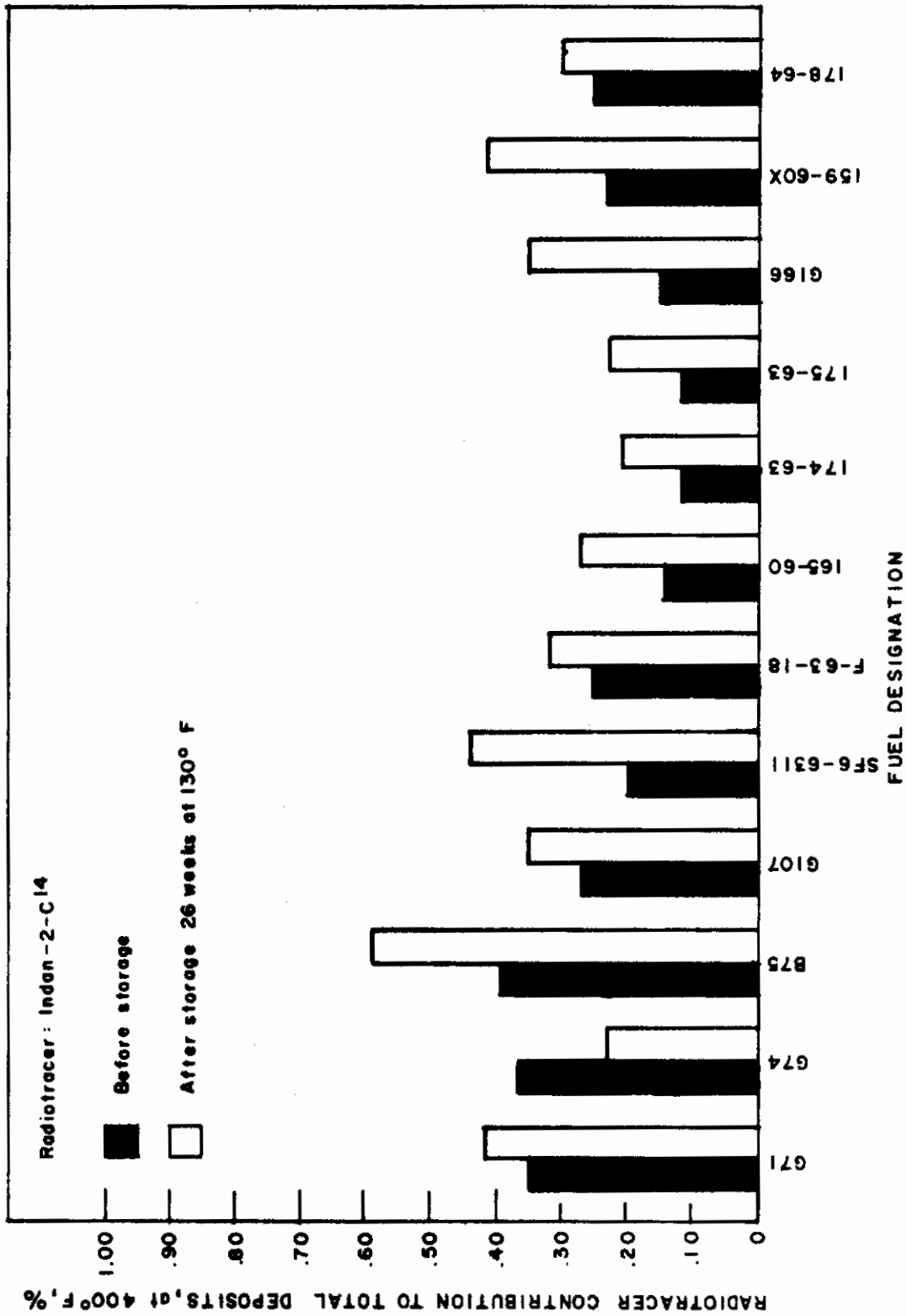


FIGURE 7. - Contribution of Indan-2-C¹⁴ to Thermally Induced Deposits at 400° F.

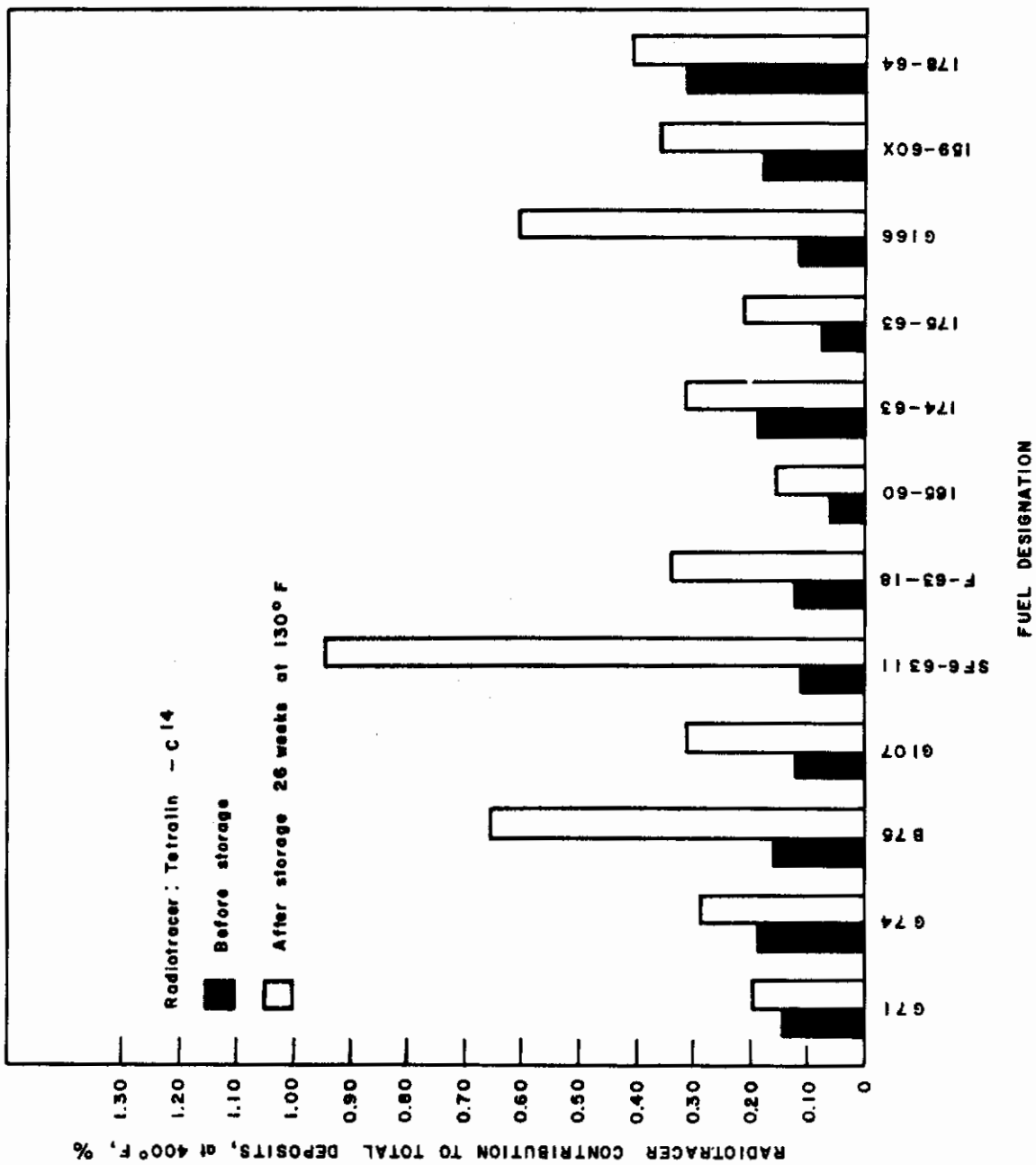


FIGURE 8. - Contribution of Tetralin-C¹⁴ to Thermally Induced Deposits at 400° F.

evident that this fuel loses much of its thermal stability quality during 26 weeks at 130° F. As mentioned earlier, this fact is evidenced by increased radiotracer contribution to deposits for practically all of the blends of -B75 tested in this investigation.

The degree of participation of a substituted naphthalene both before and after storage is shown in figure 9. The results are similar to, but slightly higher than, those observed for the benzene series in figure 5. Again fuel -B75 shows one of the largest increases after the storage interval.

Similar data from table 37 obtained for a series of blends containing a commonly used fuel antioxidant as the radiotracer are presented in figure 10. It is obvious that a different behavior is described by these data. The extent of initial contribution to deposits is much higher than for any of the systems previously discussed and the increase of participation of the labeled compound is equally dramatic for many of the stored blends. The highest levels of radioactivity were found in deposits from fuels with poor coker ratings but a good correlation is not demonstrated. However, these results raised some very important questions regarding both the mechanisms of additive degradation and the significance of these data. Even though the level of additive participation in total deposits is relatively quite high, it was reported earlier (2) that after a sample of the blend had been heated and filtered in the standard microscale bomb test, the amount of radioactivity in the sample was significantly less than the radioactivity in the untested blend. The loss in radioactivity was greater at higher test temperatures and also varied a great deal from fuel to fuel. However, there was no loss in a room temperature test. These facts suggested that heating of the blend caused part of the antioxidant to decompose or react with a component of the fuel in such a way that the butyl group, which contained the carbon-14, was lost as a volatile fragment or compound. An experimental program, therefore, was developed in an attempt to determine the identity of the carbon-14 compounds which were lost from the heated blends.

D. Thermal Decomposition Studies of di-t-Butyl-C¹⁴-p-Cresol

This study was designed with two purposes in mind. First, identification of radioactive products lost when the fuel was thermally stressed and second, a better understanding of the mechanism of this thermal decomposition.

Fuel RAF-175-63 was chosen as the base fuel for a series of experimental blends containing the cresol antioxidant. In the previous study, a similar blend showed a 25 percent loss in radioactivity after thermal stressing at 500° F.

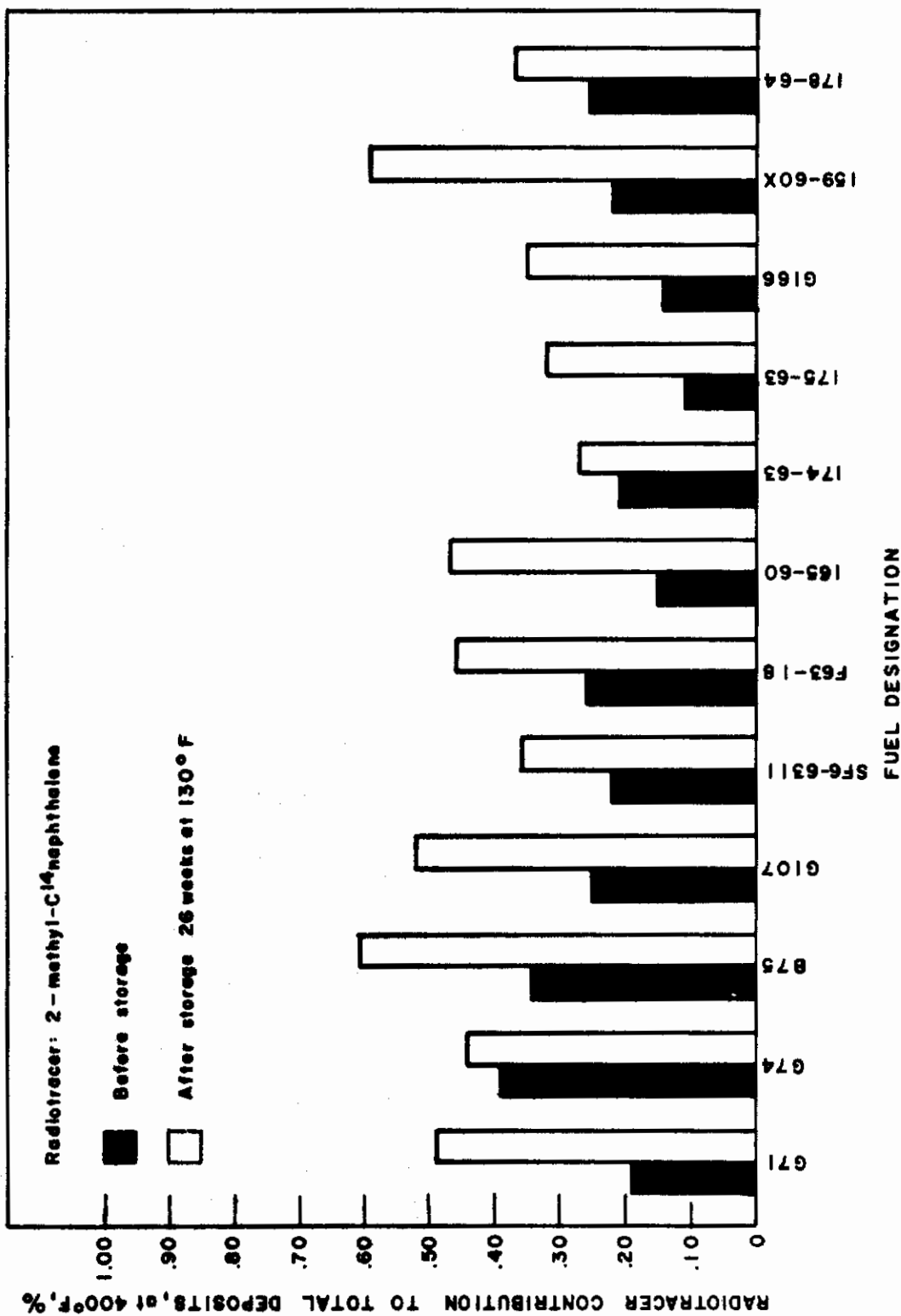


FIGURE 9. - Contribution of 2-Methyl-C¹⁴-Naphthalene to Thermally Induced Deposits at 400° F.

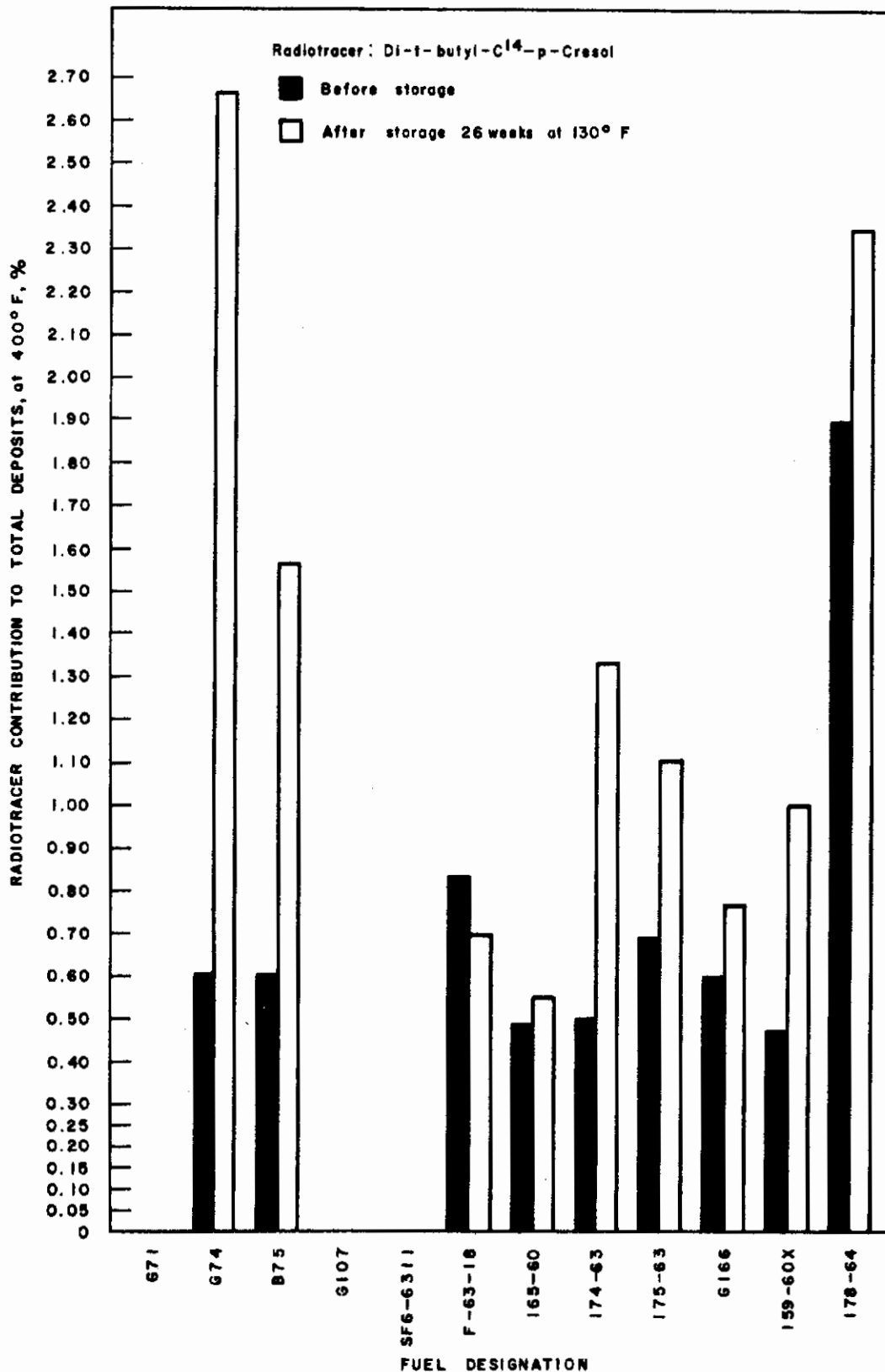


FIGURE 10. - Contribution of di-t-Butyl-C¹⁴-p-Cresol to Thermally Induced Deposits at 400° F.

Contrails

Checks were made on the gas routinely vented from the test apparatus at the end of the heating period. These gases and vapors were collected and the carbon-14 content was determined by ionization chamber current measurements using a vibrating reed electrometer. That only a very small amount of radioactivity was found in these gases indicates that most of the loss of radioactivity occurred during the vacuum filtration step of the thermal stability test procedure.

Pursuing this possibility, a trap cooled in liquid nitrogen was placed in the vacuum line between the filtration flask and the pump. The material that was collected in this trap during the filtration of a heated aliquot of the test blend was flash evaporated into a vapor phase chromatograph and separated in a 20-foot di-butyl-maleate column operated at 0° C. The results obtained on a blend containing 20 ppm of the cresol antioxidant were not conclusive so a higher level blend containing about 4250 ppm was prepared for a similar analysis.

A 5-ml aliquot of this new blend was heated to about 620° F in the microscale thermal stability bomb, then filtered through both a 0.45 μ and a 10 m μ filter with the liquid nitrogen trap in the vacuum line. Collection and subsequent chromatographic analysis of the trapped material showed two radioactive species, one much larger than the other, with possibly a third very small peak.

Another portion of this test blend was treated in a similar manner and the chromatographic peaks corresponding to the radioactive species were re-collected for mass spectral analysis. Tentative identification by the mass spectrometer was verified by checking the retention times of reference compounds corresponding to the tentative identifications. As a result of this study, identification and verification were achieved for two of the three radioactive species with tentative identification of the very small third component. The largest radioactive peak was identified as isobutene (2-methylpropene), the smaller peak as isobutane, and the smallest as n-butane.

It was observed in this study that the blend with a higher concentration of cresol additive showed much smaller losses in radioactivity than did the less concentrated blend. Equal amounts of thermal stress applied to these two blends resulted in 3-4 percent loss in the 4250 ppm blend and 25-30 percent loss in the 20 ppm blend.

Therefore, a number of blends containing different concentrations of this additive in three different base fuels were prepared. Aliquots of each were heated to about 600° F in the test apparatus and then filtered successively through a 0.45 μ and a 10 μ filter. The filtrate was radioassayed and compared with the unheated blend. The results of this study are shown in table 38. The significant observation to be made from these data is the

increase of percent radioactivity loss as the concentration of radiotracer decreases, in all three test fuels. At levels similar to the maximum allowable by MIL-J-5624E specifications, the decomposition was highest.

E. Submicron Filtration of Jet Aircraft Fuels

An additional study was performed in an attempt to delineate the action of cellulose ester filters in the filtration of labeled fuel blends. It was observed in previous experimentation that significant thermal fuel degradation often resulted in very slow filtration through the standard 10 millimicron ($m\mu$) filter. The incorporation of a prefiltration through a 0.45μ filter solved this time problem but was instrumental in uncovering another equally serious situation. It was found that only 50 percent or less of filterable deposits were removed by the first filtration through a $10 m\mu$ filter and often 3 or 4 additional filtrations were required to reduce the retained radioactivity to a constant level.

To investigate this problem a labeled fuel blend consisting of fuel RAF-175-63 plus about 10 ppm of di-t-butyl- C^{14} -p-cresol was prepared. Prior to each filtration, 5 ml of the fuel blend was placed in the microscale thermal stability bomb and pressurized to 60 psig with nitrogen. The blend was then stressed by raising the temperature continuously during a 20 minute heating cycle. The fuel temperature was approximately 420° F at the end of this heating period. Previous work had shown that significant deposits were produced at this temperature.

The filters used in this study were standard cellulose ester filters with a pore size of $10 m\mu$. After the filtration, the filters were washed with n-heptane and dissolved in 1 ml of acetone; the solution was then counted by liquid scintillation techniques. A blank run that was made on 5-ml of unheated test blend showed that filterable radioactivity was reduced to a constant minimum with one pass through a $10 m\mu$ filter. However, a second aliquot, treated and stressed as described showed that four filtrations were required to reach a constant minimum.

It was first speculated that the apparent excess of radioactive particles collected after the first filter resulted from an electrostatic phenomena. Therefore, a filter was placed in a grounded metal holder and a series of filtrations carried out as before. The use of the grounded metal filter holder did not make a significant difference in the results.

Since the fuel was exposed to atmospheric oxygen between filtrations it was also speculated that some rapid oxidation reaction might have an effect on growth of the particles. Consequently, two metal holders were connected

together, one directly above the other, so that the fuel passed through two filters without intermediate exposure to air. These results were compared directly with data obtained on another aliquot that was exposed to atmospheric oxygen for 30 minutes between each filtration. It was concluded that exposure to air did not have a short-term effect upon formation of filterable deposits.

Finally, aliquots of the heated fuel were diluted with an inert solvent prior to filtration. It was found that only about half as much material was collected during filtration as was obtained from an undiluted aliquot. It was also observed that increasing the surface area of the filter by 3.5 times resulted in about a threefold increase in filterable deposits.

In light of the foregoing investigations and data it was logical to assume that most of the radioactive particles removed by filtration are irregular in shape with one dimension greater than the pore size of the filter and another dimension smaller. The efficiency of collecting all the particles on the first filtration would be a statistical function related to particle size and filter pore size and proportional to the concentration of particles in the solution. Each succeeding filtration would remove a corresponding percentage of the particles. If only 50 percent of the existing particles were removed on each filtration, four steps would then remove over 95 percent of the particles. This corresponds well to the actual experimental data.

III. MICROHYDROGENATION WITH TRITIUM

A. General

An objective of this program was the evaluation of microhydrogenation procedures for light hydro-treatment of about 0.1 ml samples of JP-6 type fuels using hydrogen labeled or tagged with tritium. Fuel components susceptible to hydrogenation could thus be measured quantitatively by determining the total radioactivity of the treated fuel. The desired degree of hydrogenation might be obtained by varying temperature, contact time, and catalyst.

Earlier studies of a modified in situ hydrogenation technique using a tritium labeled hydrogenation reagent, although they failed to correlate with thermal stability data for a series of 12 fuels, indicated the method might be applicable to olefin determination at low levels with better sensitivity than current methods of analysis.

B. Experimental Investigations and Results

The procedure outlined by Brown et al (3), although simple and effective for bench scale hydrogenations, was found insufficiently precise and sensitive for measuring unsaturation in most high temperature aircraft fuels. In order to extend the sensitivity of this method into a range of less than 0.2 millimole olefinic unsaturation per milliliter, it was necessary to alter the method and apparatus somewhat to permit the use of tritiated sodium borohydride as a hydrogenation reagent. After the in situ hydrogenation was complete, the reaction mixture was filtered into a separatory funnel and about 150 ml of water was added to remove the alcohol. The oil phase was washed with three 20 ml portions of water before radioassay by liquid scintillation counting. This procedure was followed throughout this study with the only variation being in the time allowed for the reaction.

The first hydrogenation reagent prepared from tritiated sodium borohydride had a measured specific activity of 66 microcuries per milliliter ($\mu\text{c/ml}$). Several 1 ml samples of standard olefin blends were hydrogenated using this reagent solution and a platinum catalyst. Based upon the specific activity of the NaBH_4 solution, the final radioactivity of a 1 percent solution of octene after complete saturation should have been 10 $\mu\text{c/ml}$. The actual activity, however, was much less than this theoretical value. Both theoretical and actual amounts of tritium absorbed per unit of olefin saturation are shown in figure 11. Despite the difference between the two curves in this figure, gas phase chromatographic analysis indicated octene was completely and rapidly hydrogenated.

All of the results obtained with the platinum catalyst are listed in table 1. These data indicate that the amount of tritium incorporated varied with different olefin structures. It should be observed that the nonolefinic solvents heptane, octane, and decane all absorbed some tritium through exchange of tritium for hydrogen, resulting in high blank corrections. The two sulfur compounds that were included in table 1 absorbed very little tritium and did not seem to affect the hydrogenation of octene, as might have been expected if appreciable catalyst poisoning had occurred. Octene in octane solution produced nearly the same results as octene in decane; however, heptene in heptane produced a higher incorporation of tritium than heptene in decane.

Several results that were obtained substituting a palladium catalyst for the platinum in the hope of increasing sensitivity as well as accuracy are shown in table 2. The results were higher than those obtained with platinum and were very close to the theoretical tritium incorporation calculated from the specific activity of the hydrogenation reagent. However, new solutions of tritiated NaBH_4 hydrogenation reagent and palladium catalyst solution did not give results consistent with those in table 2. These latter data are tabulated in table 3 and graphically shown in figure 12. It is obvious that less than theoretical amounts

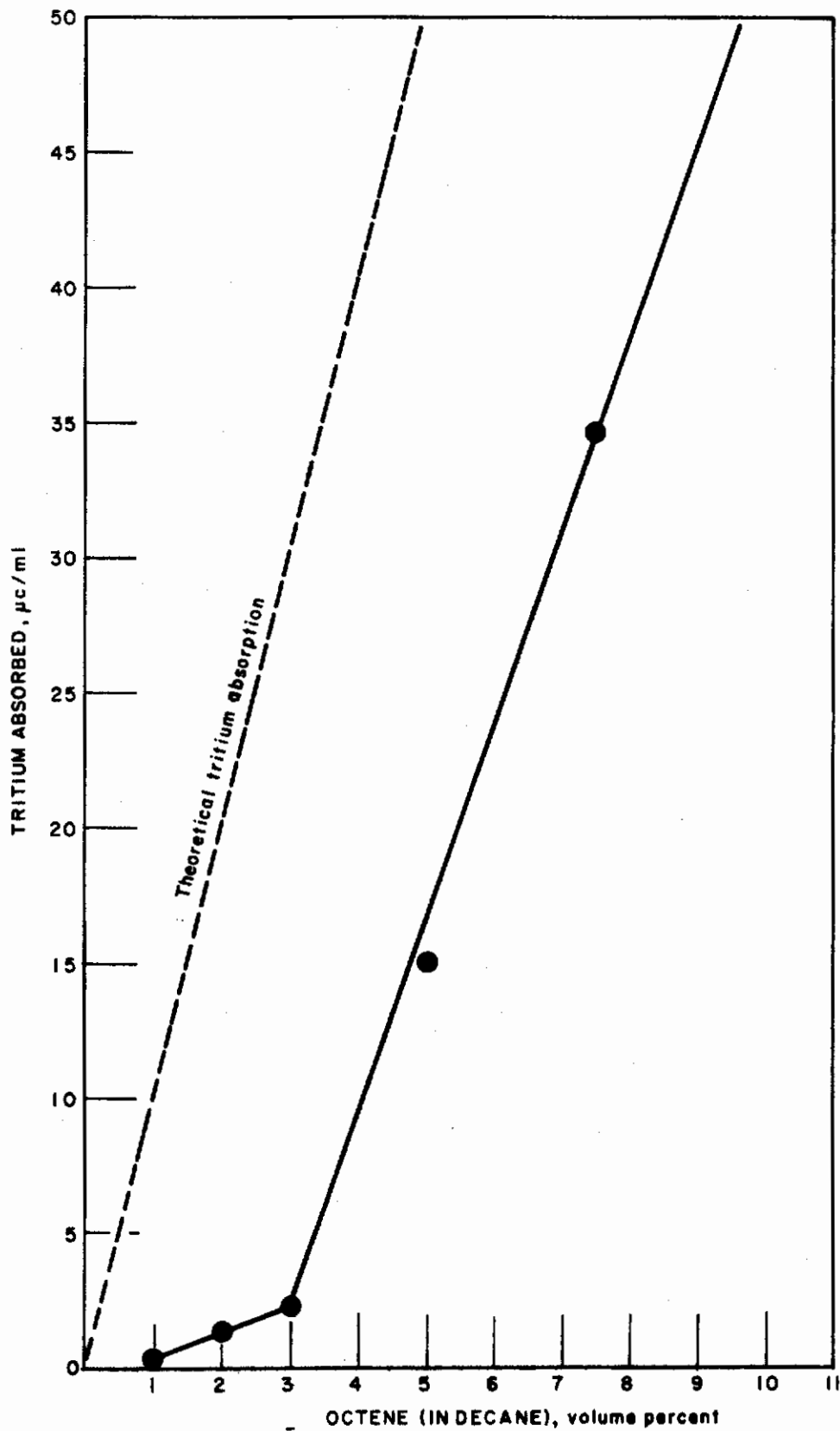


FIGURE 11. - Tritium Absorbed During Hydrogenation of Octene with a Platinum Catalyst.

TABLE 1. - HYDROGENATION OF SOME PURE OLEFINS USING A TRITIATED REAGENT AND PLATINUM CATALYST

<u>Sample</u>	<u>Unsaturation,</u> <u>mmoles/ml</u>	<u>Radioactivity</u> <u>after hydrogenation</u>		
		<u>μc/ml</u>	<u>Avg.</u>	<u>Net</u>
n-Decane	none	0.234 .264	0.249	
0.1% v Octene-1 in decane	0.00645	.291 .300	.296	0.047
0.5% v Octene-1 in decane	.0322	.339 .349	.344	.095
1% v Octene-1 in decane	.0645	.550 .425	.488	.239
2% v Octene-1 in decane	.129	1.48		1.23
3% v Octene-1 in decane	.193	2.44		2.19
5% v Octene-1 in decane	.322	14.7 15.6	15.2	15.0
7.5% v Octene-1 in decane	.484	34.9		34.7
10% v Octene-1 in decane	.645	52.4		52.2
1% v Heptene-2 in decane	.0714	.442 .436	.439	.190
2% v Heptene-2 in decane	.143	1.34 1.38	1.36	1.11
1% v Hexene-1 in decane	.0799	.432		.183
1% v Benzene in decane		.334		.085
1% v Tetralin in decane		.328		.079
1% v Indene in decane	.0866	.632		.383

TABLE 1. - HYDROGENATION OF SOME PURE OLEFINS USING A TRITIATED REAGENT AND PLATINUM CATALYST
Continued

<u>Sample</u>	<u>Unsaturation, mmoles/ml</u>	<u>Radioactivity after hydrogenation</u>		
		<u>μc/ml</u>	<u>Avg.</u>	<u>Net</u>
1% v Cyclohexene in decane	0.0986	0.474		0.225
0.1% v Cyclohexene in decane	.00986	.444		.195
1% v 1,5-Hexadiene in decane	.1676	.571		.322
1% v 4-Vinylcyclohexene-1 in decane	.1538	.690		.441
0.1% v 2-Ethylhexyl mercaptan in decane		.313		
0.1% v Ethyl disulfide in decane		.309		
1% Octene-1 plus 0.25% v ethylhexyl mercaptan in decane	.0645	.511		.262
n-Octane	none	.325		
1% v Octene-1 in octane	.0645	.598 .564	0.579	.254
5% v Octene-1 in octane	.322	19.3 20.0	19.6	19.3
n-Heptane	none	.433		
1% v Heptene-2 in heptane	.0714	.861		.428
5% v Heptene-2 in heptane	.357	12.8		12.4

TABLE 2. - HYDROGENATION OF SOME PURE OLEFINS USING A TRITIATED REAGENT AND PALLADIUM CATALYST

<u>Sample</u>	<u>Unsaturation, mmoles/ml</u>	Radioactivity after hydrogenation		
		<u>μc/ml</u>	<u>Avg.</u>	<u>Net</u>
n-Decane	none	0.988 .949	0.968	
0.1% Octene in decane	0.00645	4.59		3.62
0.5% Octene in decane	.0322	9.00		8.03
1% Octene in decane	.0645	9.98 13.38	11.68	10.71
2% Octene in decane	.129	21.0		20.0
3% Octene in decane	.193	38.8		37.8

of tritium were incorporated into these samples. Also, it can be observed that the amount of absorbed tritium appears a function of molecular weight of the olefin. This relationship probably is coincidental, however, and the significant factor may be some property such as solubility of the saturated product in water. Solubility would decrease with increasing molecular weight. With water extraction an integral part of the method it was necessary to consider solubility very carefully. Experimental testing of the role played by water solubility indicated that compounds of lower molecular weight than eight carbon atoms were soluble enough in water to have an appreciable loss during extraction.

A third solution of tritiated NaBH_4 reagent was used to hydrogenate samples of hexene in hexane and heptene in heptane for comparison with solutions of the same olefins in decane. It was found that hexene was almost completely soluble in the water and alcohol mixture that resulted from the extraction procedure. Consequently, over 90 percent of the sample was not recoverable by the extraction procedure. Heptane also was found to be soluble in water and about 70 percent of this sample was lost. These data verified to an extent previous observations regarding solubility relationship to apparent low values of hydrogenation. The data obtained with the third batch of hydrogenation reagent that was prepared are shown in table 4. Higher values comparing favorably with initial data were again obtained.

TABLE 3. - HYDROGENATION OF SOME OLEFINS USING A SECOND BATCH OF TRITIATED REAGENT AND PALLADIUM CATALYST

Sample	Unsaturation, mmoles/ml	Radioactivity after hydrogenation	
		$\mu\text{c/ml}$	Net
n-Decane	none	0.7563 .6946	Average = 0.725
1% v Hexene-1 in decane	0.0799	1.44	0.72
2% v Hexene-1 in decane	.1598	3.37	2.65
1% v Heptene-2 in decane	.0714	3.41 3.26	2.69 2.54
2% v Heptene-2 in decane	.143	6.42	5.70
0.1% v Octene-1 in decane	.00645	1.85	1.13
0.5% v Octene-1 in decane	.0822	3.10	2.38
1% v Octene-1 in decane	.0645	4.43 4.45 4.39	3.71 3.73 3.67
2% v Octene-1 in decane	.129	7.20	6.48
3% v Octene-1 in decane	.193	11.80	11.08
5% v Octene-1 in decane	.322	23.4	22.7
1% v Benzene in decane		.771	.046
1% v Tetralin in decane		5.52	4.80
1% v Indene in decane	.0866	2.73	2.01
1% Cyclohexene in decane	.0986	2.45	1.73

TABLE 3. - HYDROGENATION OF SOME OLEFINS USING A SECOND
 BATCH OF TRITIATED REAGENT AND PALLADIUM
 CATALYST
 Continued

<u>Sample</u>	<u>Unsaturation, mmoles/ml</u>	<u>Radioactivity after hydrogenation</u>	
		<u>μc/ml</u>	<u>Net</u>
0.1% v Cyclohexene	0.00986	0.973	0.248
1% v 1,5-Hexadiene in decane	.1676	3.55	2.83
1% 4-Vinylcyclohexene-1 in decane	.1538	10.42	9.70
n-Octane	none	1.19	
0.1% v Octene-1 in octane	.00645	2.30	1.11
0.5% v Octene-1 in octane	.0322	3.86	2.67
1% v Octene-1 in octane	.0645	4.63	3.44
2% v Octene-1 in octane	.129	7.88	6.69

This study, although inconclusive in itself, pointed out the need for a revision of procedure, particularly the extraction step for sample recovery. High blank values for non-olefinic compounds such as heptane, octane, and decane diminished the promise for the method and lack of good sensitivity at low levels of radioactivity in the hydrogenation reagent was equally disappointing. Finally, the poor precision obtained during the course of these experiments made continuation of the study inadvisable.

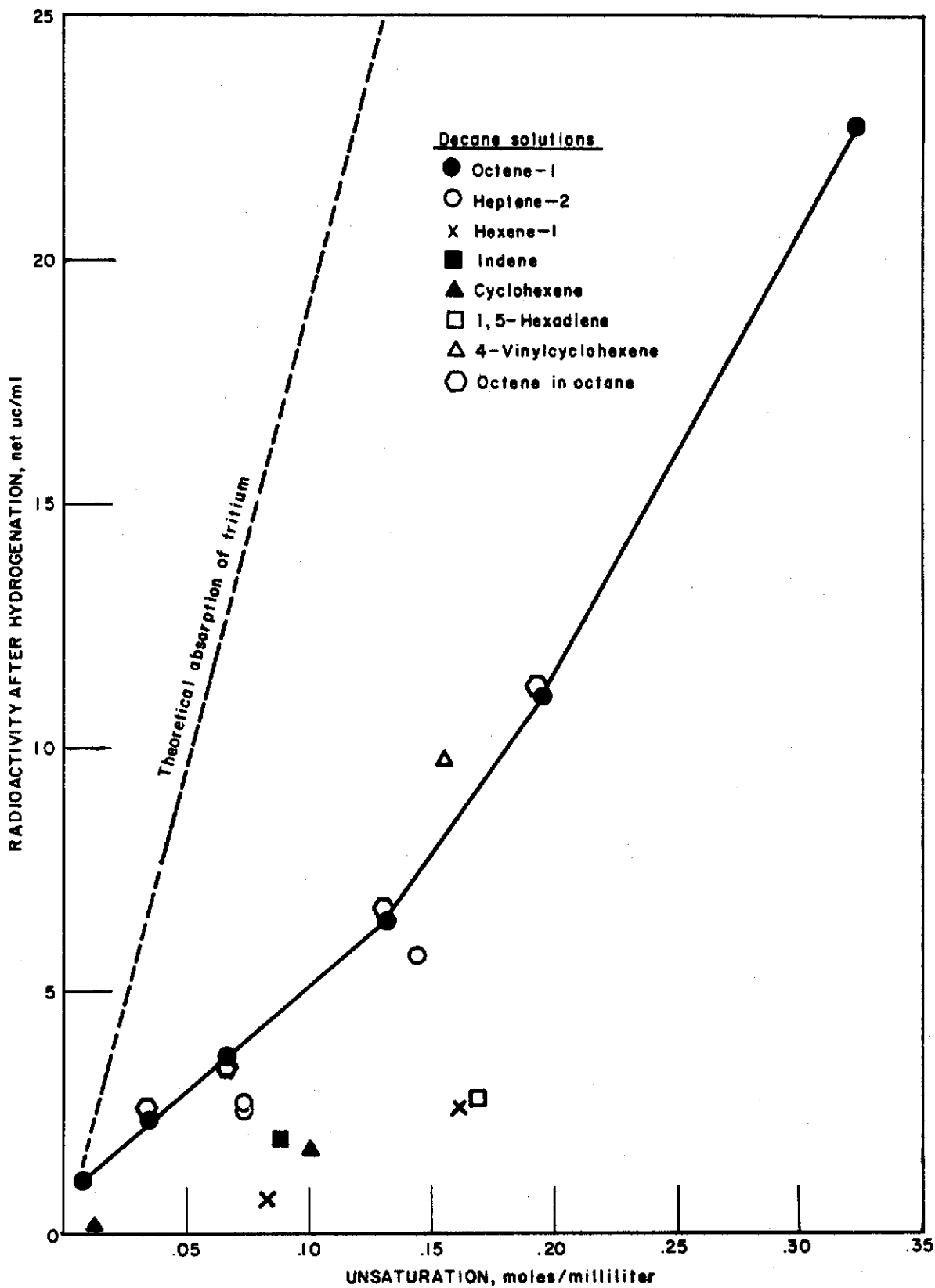


FIGURE 12. - Radioactivity of Samples Hydrogenated Using a Second Batch of Tritiated Reagent and Palladium Catalyst.

TABLE 4. - HYDROGENATION USING A THIRD BATCH OF TRITIATED REAGENT AND PALLADIUM CATALYST

<u>Sample</u>	<u>Mmoles of Unsaturation</u>	<u>Activity after Hydrogenation</u>	<u>Net $\mu\text{c/ml}$</u>
Decane "blank"	none	1.05 $\mu\text{c/ml}$	
1% Octene in decane	0.0645	9.72	8.67
1% Hexene in decane	.0799	2.96	1.91
2% Hexene in decane	.1598	11.87	10.82
Hexane "blank"		none recovered	
1% Hexene in hexane	.0799	14.88	
2% Hexene in hexane	.1598	none recovered	
1% Heptene in decane	.0714	10.08	9.03
2% Heptene in decane	.1428	24.06	23.01
Heptane "blank"		13.49	
1% Heptene in heptane	.0714	36.44	22.95
2% Heptene in heptane	.1428	42.89	29.40

IV. INITIATION OF FUTURE PROGRAMS

It is anticipated that this project will be extended to utilize a microcoker for dynamic thermal stability tests on fuels to which small amounts of labeled compounds have been added. A number of the appropriate C¹⁴ labeled compounds that are needed for this investigation have been obtained. Included in these materials are carbon-14 labeled 3-methylindene, 3-ethylindene, 1-methylindan, and 1-ethylindan. These labeled materials were custom synthesized by a Canadian supplier of carbon-14 labeled compounds.

It is essential that these materials be repurified before using them in a future program to investigate thermally induced deposits. This repurification is necessary after a period of only a few weeks because of self decomposition promoted by release of high energy beta particles through radioactive decay. This problem does not exist in blends to which the radiotracers have been added because of the dilution of small amounts of radioactivity by large volumes of fuel. Reference materials for use in gas phase chromatographic purification of the above materials were not available, and therefore it has been necessary to develop procedures for synthesis of many of these reference materials.

The exact methods that were used to prepare these indenenes and indans can be found in Appendix IV of this report. The essential steps include a Grignard reaction of an appropriate ketone. The product of this reaction is hydrolyzed to obtain the corresponding indanol, followed by a dehydration procedure with acid to the indene. An aliquot of the indene is subsequently hydrogenated with Raney nickel catalyst to obtain the corresponding indan.

All of the alkylated indenenes and indans that are scheduled for investigation are now on hand as a result of this synthesis program. Procedures are currently being developed for the preparation of carbon-14 labeled additives that are also scheduled for study in a future program.

It is planned to measure the contribution of the labeled compounds to microcoker tube deposits, so a proportional counter has been designed, built, and tested for measuring the radioactivity in deposits on the microcoker tube.

V. CONCLUSIONS

A. Microscale Thermal Stability Deposits

A radiotracer technique was successfully developed that extended the sensitivity of determinations to the parts-per-billion range for contributors to deposit formation in stored and thermally stressed jet fuel. This method was

evaluated by application to 88 test blends, representing 12 fuels and 8 carbon-14 labeled fuel constituents and 1 fuel additive. Among the materials tested, aromatic compounds were found to be the largest contributors to total deposits determined by this method. Five aromatic materials were tested and tetralin showed the largest contribution to deposits, closely followed by 2-methyl naphthalene and indan. Paraffinic and olefinic compounds were smaller contributors to storage and thermally induced varnishes and deposits.

One fuel additive evaluated by this method was a cresol-type antioxidant. More than 2.0 percent of this additive reacted or decomposed and appeared as a thermally induced deposit in two unstable fuels. In addition, another 25 percent of the additive was lost through thermal decomposition at temperatures above 400° F. Volatile products of this thermal decomposition were identified as isobutene, with lesser amounts of isobutane and n-butane. These compounds resulted from fragmentation of the butyl group from the antioxidant. Thermal decomposition of this additive was more pronounced after the blend had been stored for 26 weeks at 130° F. However, significant variations in reaction and thermal decomposition were noted between fuel matrices. In general, the more thermally unstable fuels promoted the largest losses of additive. It also was observed that the extent of thermal decomposition of this additive was controlled by its concentration in the fuel matrix. At levels similar to the maximum allowable by MIL-J-5624E fuel specifications, the decomposition was largest while at much higher concentrations the percentage loss decreased. It was concluded that storage followed by relatively short periods of thermal stress can seriously deplete and diminish the effectiveness of this antioxidant. This study serves to point out the need of further research into high temperature fuel additives with the objective of evaluation in dynamic rather than in static systems. The rapid depletion of fuel additives in coker-type test apparatus might indicate the need for fuel antioxidants with higher resistance to thermal degradation.

Another conclusion reached in this study was that a single filtration through submicron cellulose filters is often less than 50 percent effective in removing all particles and a third and even fourth filtration were found necessary to reduce filterable deposits to a constant minimum.

B. Microhydrogenation With Tritium

A series of shelf purity olefins were hydrogenated in the evaluation of a modified in situ hydrogenation technique using a tritium labeled reagent. The goal of this work was to increase and extend the sensitivity and accuracy of determination for low levels of olefins in high temperature fuels. A comparison of the extent of tritium absorption in standard blends of olefinic samples showed that a palladium catalyst was more effective than a platinum catalyst in achieving theoretical levels of radioactivity in hydrogenated products. Each catalyst, however, gave complete saturation of double bonds as determined by vapor phase chromatography. It was noted that separately

prepared batches of tritiated hydrogenation reagent produced different levels of tritium incorporation, although precision within a single batch was good. Still another conclusion concerned the sample recovery after hydrogenation. It was found that preferential solubility in water used for sample extraction caused wide variation in results. Lower molecular weight hydrocarbons were very soluble. This circumstance led to a high source of error in the determination of olefins of less than eight carbon atoms. Finally it was concluded that relatively high levels of radioactivity were required for the determination at an acceptable level of sensitivity. The combination of these factors, plus the sophisticated radioassay equipment required, limited the practical application of the technique to a large extent and was instrumental in the decision to discontinue this line of endeavor.

VI. RECOMMENDATIONS

Information obtained and described in this report indicates that extensions of some of these procedures offer the most promising techniques for determining, predicting, and possibly understanding the thermal stability phenomena associated with high temperature jet fuels. The microscale thermal stability test that was developed and evaluated extended the sensitivity of determinations to the parts-per-billion range for contributors to deposit formation in stored and thermally stressed jet fuels. However, since time as well as temperature affects deposit formation, results obtained in a static system do not correspond nor correlate readily with results obtained in a dynamic system where the time of stressing is relatively short. Therefore the type of data that has been obtained and presented in this study should be extended using a microcoker apparatus that more closely simulates aircraft fuels systems in supersonic flight. The microcoker should be utilized to test fuels that have been labeled with radiotracers to determine the potential usefulness of the technique to predict thermal stability quality during storage. The method would be designed such that radioactivity incorporated into deposits on the preheater test section and the test filters would be measured for correlative application.

Using the same test method, another study should be inaugurated to define fuel components that contribute significantly to deposits. With the verification of these components, a more comprehensive program of reaction mechanisms should be designed as follows:

1. It would be determined if the labeled deposit-former, when present in an unstable fuel environment, was significantly affected by clay, activated carbon, or gel percolation. Factors that could be significant in this investigation would include removal of the labeled component by the adsorption

Contrails

treatment or the removal of polar material and other fuel constituents that are essential to the reaction mechanisms involving the labeled component.

2. Determine, by thermally stressing the test blend, the effect of added contaminants such as water, dissolved metals, oxygen, and others upon the rate and extent of reaction of the labeled fuel component introduced.
3. Evaluate the effect of ambient storage upon all of the above factors. Storage tests previously performed in this program indicate that 6 months' storage at 130° F has little or no effect upon the thermal stability of many high temperature fuels. These tests will be augmented by longer periods of storage at elevated temperatures.

In previous efforts with the microscale thermal stability bomb, some types of compounds gave indications of contributing to fuel system deposits. In a future program, these compounds would be labeled with radiotracers and tested in the microcoker. In this phase of the program, the labeled components to be evaluated in fuels should include, but not necessarily be limited to, the following:

- (1) n-Hendecane-1-C¹⁴
- (2) Diphenyl-C¹⁴
- (3) 1,2,3,5-Tetramethylbenzene-C¹⁴
- (4) 1-Dodecene-1-C¹⁴
- (5) Tetralin-C¹⁴
- (6) 3-Methylindene-C¹⁴
- (7) 3-Ethylindene-C¹⁴
- (8) 1-Methylindan-C¹⁴
- (9) 1-Ethylindan-C¹⁴
- (10) 2-Methyl-C¹⁴-naphthalene

Previous results using radiotracer techniques for studying fuel additives should be extended to include the following:

- (1) Antioxidants
- (2) Metal deactivators
- (3) Corrosion inhibitors
- (4) Experimental additives

The rate of loss of these additives in several fuel environments at high temperatures would be determined and efforts made to identify thermally induced degradation products of these additives. The results of this study should be aimed at providing information regarding the suitability of present and future jet fuel additives, verifying, if possible, the mechanisms by which these additives accomplish their purposes.

The carbon-14 labeled fuel additives of this study should be investigated in a series of five fuels, including the following types:

- (1) JP-5 (MIL-T-5624)
- (2) JP-6 (MIL-J-25656)
- (3) Thermally stable (MIL-F-25524)
- (4) Experimental high temperature fuels.

In addition, polar materials could be removed from a few of these fuels by percolation over clay or carbon to determine their effect upon additive consumption under severe stress conditions.

Fuels treated for removal of polar-type fuel components would be stored for retesting to evaluate the effectiveness of additives in preventing peroxide formation.

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APPENDIX I
TEST FUELS AND SPECIAL
BLENDS OBTAINED AND
PREPARED FOR THIS PROGRAM

APPENDIX I

FUELS AND TEST BLENDS

1. Test Fuels

Twelve fuels were obtained in 1 quart amber bottles for initial studies. These fuels were stored at freezer chest temperatures and opened only at room temperatures to minimize possibilities of moisture condensation inside the bottle. As studies have proceeded during this reporting period, some of the original quart samples have become depleted and additional 1 gallon samples were obtained. The numbers which were assigned these fuels and other information supplied are shown in table 5.

In addition to samples listed in table 5, an additional seventeen 1 quart samples have been obtained bearing numbers BJ-64-10-G213 through G229. No test data are currently available on these latter fuels.

2. Test Blends

Table 6 is a tabulation of pertinent information regarding 94 test blends that have been prepared for testing in the small scale static thermal stability apparatus. These blends represent a combination of 9 labeled fuel components and 12 test fuels in concentrations ranging from 1 to 20 ppm of the radiotracer.

TABLE 5

FUELS CURRENTLY ON HAND

Fuel Designation	Other Identifying Numbers	Threshold Failure Temp., °F			Calculated Temperature, °F, for 25 Units Δ LT Loss	
		ASTM-CRC			Phillips	Bureau of Mines
		SSF Coker	Coker	Minex		
RAF-178-64	BJ-64-10-G201		300			383
-G74	BJ-64-10-G74	332	360		395	383
SF6-6311	BJ-64-10-K147		450		568	437
-G166	BJ-64-10-G166	425	N/A		466	437
RAF-159-60X	BJ-64-10-L200		N/A		523	547
RAF-175-63	BJ-64-10-G163		435	460	521	433
-G71	BJ-64-10-G71	712	450+		558	411
RAF-165-60	BJ-64-10-G158		375-400			462
-G107	BJ-64-10-G107	692	450+		566	435
-B75	BJ-63-10-G75	625	500		626	438
RAF-174-63	BJ-64-10-G162		360	405	384	378
F-63-18	BJ-64-10-K148		N/A	575	576	451

TABLE 6

TEST BLENDS PREPARED FOR RADIOTRACER METHOD DEVELOPMENT

Test Blend	Fuel	Radiotracer	Concentration, ppm ^{1/}	Sp. Act. μc/ml
RR-C ¹⁴ -746	RAF-165-60	Diphenyl-C ¹⁴	9	0.0322
RR-C ¹⁴ -747	RAF-174-63	do.	9	.0384
RR-C ¹⁴ -748	BJ-64-10-G74	do.	9	.0404
RR-C ¹⁴ -749	F-63-18	do.	9	.0386
RR-C ¹⁴ -750	RAF-175-63	do.	9	.0395
RR-C ¹⁴ -754	BJ-64-10-G166	di-t-Butyl-C ¹⁴ -p-cresol	20	.0156
RR-C ¹⁴ -755	RAF-165-60	do.	20	.0215
RR-C ¹⁴ -756	BJ-64-10-G74	do.	20	.0423
RR-C ¹⁴ -757	F-63-18	do.	20	.0823
RR-C ¹⁴ -764	RAF-175-63	do.	20	.0734
RR-C ¹⁴ -766	RAF-165-60	do.	20	.0559
RR-C ¹⁴ -772	RAF-178-64	do.	20	.0773
RR-C ¹⁴ -773	BJ-63-10-B75	do.	20	.0698
RR-C ¹⁴ -774	RAF-175-63	do.	20	.0776
RR-C ¹⁴ -776	RAF-159-60X	do.	20	.0349
RR-C ¹⁴ -784	RAF-165-60	1-Dodecene-1-C ¹⁴	2	.0166
RR-C ¹⁴ -786	RAF-174-63	do.	2	.0217
RR-C ¹⁴ -787	BJ-63-10-B75	do.	2	.0254
RR-C ¹⁴ -804	BJ-64-10-G71	do.	2	.0265
RR-C ¹⁴ -806	RAF-178-64	Tetralin-C ¹⁴	2	.0691
RR-C ¹⁴ -807	BJ-64-10-G74	do.	2	.0529
RR-C ¹⁴ -808	BJ-64-10-G71	do.	2	.0593
RR-C ¹⁴ -810	RAF-175-63	do.	2	.0600
RR-C ¹⁴ -811	SF6-6311	do.	2	.0577
RR-C ¹⁴ -813	BJ-64-10-G166	do.	2	.0640
RR-C ¹⁴ -814	BJ-64-10-G107	do.	2	.0673
RR-C ¹⁴ -815	RAF-159-60X	do.	2	.0603
RR-C ¹⁴ -816	SF6-6311	do.	2	.0406
RR-C ¹⁴ -817	BJ-63-10-B75	do.	2	.0667
RR-C ¹⁴ -820	F-63-18	do.	2	.0640
RR-C ¹⁴ -822	RAF-165-60	do.	2	.0615
RR-C ¹⁴ -830	RAF-174-63	do.	2	.0589

^{1/} Approximate value based upon suppliers' data.

TABLE 6

TEST BLENDS PREPARED FOR RADIOTRACER METHOD DEVELOPMENT
Continued

Test Blend	Fuel	Radiotracer	Concentration, ppm ^{1/}	Sp. Act. μc/ml
RR-C ¹⁴ -831	BJ-64-10-G74	1-Dodecene-1-C ¹⁴	2 (est.)	0.0098
RR-C ¹⁴ -832	BJ-64-10-G107	do.	6 (est.)	.0314
RR-C ¹⁴ -833	BJ-64-10-G166	do.	6 (est.)	.0310
RR-C ¹⁴ -834	SF6-6311	do.	6 (est.)	.0324
RR-C ¹⁴ -835	F-63-18	do.	6 (est.)	.0316
RR-C ¹⁴ -837	RAF-175-63	do.	6 (est.)	.0316
RR-C ¹⁴ -838	RAF-159-60X	do.	6 (est.)	.0290
RR-C ¹⁴ -839	RAF-178-64	do.	6 (est.)	.0320
RR-C ¹⁴ -841	F-63-18	2-Methyl-C ¹⁴ -naphthalene	1	.0352
RR-C ¹⁴ -842	BJ-64-10-G107	do.	1	.0348
RR-C ¹⁴ -843	RAF-174-63	do.	1	.0355
RR-C ¹⁴ -844	RAF-159-60X	do.	1	.0364
RR-C ¹⁴ -853	BJ-64-10-G74	do.	1	.0331
RR-C ¹⁴ -854	SF6-6311	do.	1	.0360
RR-C ¹⁴ -855	BJ-64-10-G166	do.	1	.0347
RR-C ¹⁴ -856	RAF-175-63	do.	1	.0383
RR-C ¹⁴ -857	BJ-63-10-B75	do.	1	.0374
RR-C ¹⁴ -858	RAF-165-60	do.	1	.0370
RR-C ¹⁴ -859	BJ-64-10-G71	do.	1	.0383
RR-C ¹⁴ -860	RAF-178-64	do.	1	.0349
RR-C ¹⁴ -862	BJ-63-10-B75	1,2,3,5-Tetramethylbenzene- C ¹⁴	3	.0275
RR-C ¹⁴ -874	RAF-174-63	do.	3	.0276
RR-C ¹⁴ -875	RAF-175-63	do.	3	.0250
RR-C ¹⁴ -876	BJ-64-10-G107	do.	3	.0251
RR-C ¹⁴ -877	F-63-18	do.	3	.0275
RR-C ¹⁴ -878	RAF-159-60X	do.	3	.0252
RR-C ¹⁴ -879	SF6-6311	do.	3	.0270
RR-C ¹⁴ -880	BJ-64-10-G166	do.	3	.0276
RR-C ¹⁴ -881	BJ-64-10-G71	do.	3	.0247
RR-C ¹⁴ -882	RAF-165-60	do.	3	.0245
RR-C ¹⁴ -883	BJ-64-10-G74	do.	3	.0266
RR-C ¹⁴ -885	RAF-178-64	do.	3	.0286
RR-C ¹⁴ -899	BJ-63-10-B75	Indan-2-C ¹⁴	2	.0182
RR-C ¹⁴ -900	BJ-64-10-G74	do.	3	.0250
RR-C ¹⁴ -901	RAF-165-60	do.	3	.0258
RR-C ¹⁴ -902	RAF-175-63	do.	3	.0255

^{1/} Approximate value based upon suppliers' data.

TABLE 6
TEST BLENDS PREPARED FOR RADIOTRACER METHOD DEVELOPMENT
Continued

Test Blend	Fuel	Radiotracer	Concentration, ppm ^{1/}	Sp. Act. μc/ml
RR-C ¹⁴ -903	RAF-174-63	Indan-2-C ¹⁴	3	0.0264
RR-C ¹⁴ -904	RAF-159-60X	do.	3	.0276
RR-C ¹⁴ -905	SF6-6311	do.	3	.0259
RR-C ¹⁴ -906	BJ-64-10-G71	do.	3	.0268
RR-C ¹⁴ -907	BJ-64-10-G166	do.	3	.0260
RR-C ¹⁴ -909	F-63-18	do.	3	.0266
RR-C ¹⁴ -910	BJ-64-10-G107	do.	3	.0270
RR-C ¹⁴ -911	RAF-178-64	do.	3	.0266
RR-C ¹⁴ -925	RAF-165-60	n-Hendecane-1-C ¹⁴	2.5	.0151
RR-C ¹⁴ -926	RAF-175-63	do.	2.5	.0315
RR-C ¹⁴ -927	BJ-64-10-G107	do.	2.5	.0271
RR-C ¹⁴ -928	BJ-64-10-G166	do.	2.5	.0304
RR-C ¹⁴ -929	RAF-175-63	do.	2.5	.0281
RR-C ¹⁴ -930	RAF-159-60X	do.	2.5	.0302
RR-C ¹⁴ -931	BJ-64-10-G71	do.	2.5	.0291
RR-C ¹⁴ -932	BJ-63-10-B75	do.	2.5	.0275
RR-C ¹⁴ -933	SF6-6311	do.	2.5	.0254
RR-C ¹⁴ -934	F-63-18	do.	2.5	.0301
RR-C ¹⁴ -935	BJ-64-10-G74	do.	2.5	.0289
RR-C ¹⁴ -936	RAF-178-64	do.	2.5	.0280
RR-C ¹⁴ -955	F-63-18	di-t-Butyl-C ¹⁴ -p-cresol	20	.0214
RR-C ¹⁴ -956	RAF-175-63	do.	20	.0264
RR-C ¹⁴ -957	RAF-178-64	do.	20	.0120
RR-C ¹⁴ -959	BJ-64-10-G74	do.	20	.0194
RR-C ¹⁴ -960	BJ-64-10-G107	1,5-Hexadiene-1,6-C ¹⁴	7	.0378
RR-C ¹⁴ -961	BJ-64-10-G166	do.	5	.0247

^{1/} Approximate value based upon suppliers' data.

APPENDIX II
OPERATIONAL PROCEDURE FOR
MICROSCALE THERMAL STABILITY
APPARATUS AND PROPORTIONAL
COUNTER

APPENDIX II

MICROSCALE THERMAL STABILITY BOMB-PROPORTIONAL COUNTER

General Operational Procedure

The general method used for obtaining and measuring deposits inside the 5-ml bomb was as follows:

The temperature- ΔT relationship for the fuel was first established using an unlabeled sample. Subsequent tests with labeled test blends were performed at a temperature sufficiently high to show significant changes in light transmittance at 350 m μ ; one about 25 units; and the final, about 40 units loss. The test blend which contained a pure compound labeled with a low-energy beta emitter was first filtered through a 0.45 micron Millipore filter. An aliquot of this filtered material was then assayed by liquid scintillation counting to determine the specific activity of the sample for subsequent calculations.

A background count was made for at least 10 minutes upon the clean microbomb to be used for the ensuing analysis. Any counts above normal background (15-25 cpm) were regarded as contamination, and efforts were made to remove them by electropolishing cleaning procedures (4).

Five ml of the test blend were pipetted into the microbomb and pressured to 60 psig with nitrogen for 20 minutes. At the end of this time, the bomb was vented and the sample discarded. The bomb was carefully rinsed with n-heptane and acetone and dried with a gentle stream of air.

Another background count was then obtained using identical proportional counting conditions. The difference between these two background counts was directly attributed to adsorption and not reaction products, and subsequent calculations took this difference into account.

Finally, 5 ml of the test blend was pipetted into the bomb, and the thermocouple assembly was installed. The apparatus was pressured to 60 psig with nitrogen and inserted into the furnace which had been preheated to 90° F. The furnace power was set to give a preselected ΔT , and after 20 minutes the temperature of the fuel was recorded and the microbomb was removed from the furnace and cooled in ice water to 90° F.

The thermocouple assembly was removed from the microbomb cup, and the fuel was poured into a B&L Spectronic 20 test tube. After 45 minutes the transmittance at 350 m μ was measured. (The spectrophotometer was standardized

with isooctane). The percent transmittance of the fresh blend was compared to that of the heated fuel to obtain the ΔT .

The fuel was filtered through a 10 m μ Millipore filter. The filter was rinsed with n-heptane and then dissolved in 1 cc of acetone preparatory to radioassay by liquid scintillation counting. In the event more than 1 hour was required to pass the fuel through a 10 m μ filter, it was first prefiltered through a 0.45 micron filter.

The microbomb was rinsed twice with n-heptane and dried with a gentle stream of air. The radioactivity adhering to the inside walls of the microbomb was determined by proportional counting.

The proportional counting procedure using the microbomb was as follows:

Proportional counting gas (argon - 90 percent and methane - 10 percent) was passed through the counter at about 20 cc/min. The high voltage applied across the counter was 2,200 volts. A discriminator setting was employed to cut out some low-energy response thereby improving the background count.

The counter was flushed with the counting gas for 10 minutes before counting was started. Sufficient total counts were taken to reduce the counting error ($\sqrt{n}/n \times 100$, where n = total number of counts) caused by the random nature of radioactive decay to an acceptable level (1-2 percent).

The bomb was cleaned by electropolishing (4) before subsequent tests.

APPENDIX III

TEST DATA OBTAINED FROM RADIOTRACER STUDIES

WITH MICROSCALE THERMAL STABILITY

APPARATUS

TABLE 7

SMALL SCALE THERMAL STABILITY TEST DATA FOR TEST BLEND RR-C¹⁴-899

Fuel: BJ-63-10-B75
Labeled Component: Indan-2-C¹⁴

Sp. Act.: 0.01815 $\mu\text{c/ml}$
Total Act.: .09075 $\mu\text{c/5 ml}$

Test No.	Before Storage				After 26 wks. at 130° F			
	1	2	3	4	1	2	3	4
Avg. % T before Testing <u>1/</u>	60.2				38.4			
Final Test Temp., °F	room	393	499	622	room	401	502	622
Avg. % T after Testing <u>1/</u>	60.5	44.5	37.2	32.2	38.5	22.0	9.5	5.8
Δ LT <u>2/</u>		15.7	23.0	28.0		16.4	28.9	32.6
Adherent Deposits, dpm	0	10	0	15	0	25	30	55
μc		0.00000		0.00001		0.00001	0.00001	0.00003
%				.00		.01	.01	.03
Filterable Deposits, dpm	303	795	513	385	458 ^{3/} 860 ^{4/}	404 ^{3/} 768 ^{4/}	359 ^{3/} 915 ^{4/}	431 ^{3/} 835 ^{4/}
μc	0.00014	.00036	0.00023	.00017	0.00020	.00018	.00016	.00019
%	.15	.39	.25	.19	.23	.20	.18	.21
Total Deposits, μc	.00014	.00036	.00023	.00018	.00059	.00054	.00058	.00060
%	.15	.39	.25	.19	.65	.59	.64	.65

1/ Light transmission at 350 m μ .

2/ Light transmittance loss.

3/ Required prefiltering through 0.45 micron filter.

4/ Filtrate from 0.45 μ filter passed through 10 m μ filter.

TABLE 8

SMALL SCALE THERMAL STABILITY TEST DATA FOR TEST BLEND RR-C¹⁴-900

Fuel: BJ-64-10-G74
Labeled Component: Indan-2-C¹⁴

Sp. Act.: 0.02504 $\mu\text{c/ml}$
Total Act.: .12520 $\mu\text{c/5 ml}$

Test No.	Before Storage				After 26 wks. at 130° F			
	1	2	3	4	1	2	3	4
Avg. % T before Testing <u>1/</u>	96.9				8.0			
Final Test Temp., °F	room	364	390	396	room	363	377	401
Avg. % T after Testing <u>1/</u>	97.0	91.2	74.5	45.0	8.5	8.8	9.0	6.2
Δ LT <u>2/</u>		5.7	22.4	51.9				
Adherent Deposits, dpm	0	10	0	0	0	10	5	40
μc		0.00000				0.00000	0.00000	0.00002
%								.01
Filterable Deposits, dpm	242	540	278	687 ^{3/} 3234 ^{4/}	364 ^{3/} 2244 ^{4/}	414 ^{3/} 1924 ^{4/}	330 ^{3/} 2114 ^{4/}	354 ^{3/} 2394 ^{4/}
μc	0.00011	.00024	0.00013	0.00031	0.00016	.00019	.00015	.00016
%	.09	.19	.10	.25	.14	.15	.12	.13
Total Deposits, μc	.00011	.00024	.00013	.00045	.00026	.00028	.00025	.00029
%	.09	.19	.10	.37	.22	.22	.20	.23

1/ Light transmission at 350 m μ .

2/ Light transmittance loss.

3/ Required prefiltering through 0.45 micron filter.

4/ Filtrate from 0.45 μ filter passed through 10 m μ filter.

TABLE 9

SMALL SCALE THERMAL STABILITY TEST DATA FOR TEST BLEND RR-C¹⁴-901

Fuel: RAF-165-60
Labeled Component: Indan-2-C¹⁴

Sp. Act.: 0.02575 $\mu\text{c/ml}$
Total Act.: .12875 $\mu\text{c/5 ml}$

Test No.	Before Storage				After 26 wks. at 130° F			
	1	2	3	4	1	2	3	4
Avg. % T before Testing ^{1/}	52.8				53.2			
Final Test Temp., °F	room	402	477	621	room	402	479	621
Avg. % T after Testing ^{1/}	53.0	39.0	27.2	19.5	53.0	40.0	28.5	20.2
Δ LT ^{2/}		13.8	25.6	33.3		13.2	24.7	33.0
Adherent Deposits, dpm	15	0	10	0	0	0	0	0
μc	0.00001		0.00000					
%	.00							
Filterable Deposits, dpm	401	401	772	360	308 ^{3/} 272 ^{4/}	493 ^{3/} 288 ^{4/}	1335 ^{3/} 353 ^{4/}	446 ^{3/} 316 ^{4/}
μc	.00018	0.00018	.00035	0.00016	0.00014	0.00022	0.00060	0.00020
%	.14	.14	.27	.12	.00012 .11 .10	.00013 .17 .10	.00016 .47 .12	.00014 .16 .11
Total Deposits, μc	.00019	.00018	.00035	.00016	.00026	.00035	.00076	.00034
%	.14	.14	.27	.12	.21	.27	.59	.27

- ^{1/} Light transmission at 350 m μ .
- ^{2/} Light transmittance loss.
- ^{3/} Required prefiltering through 0.45 micron filter.
- ^{4/} Filtrate from 0.45 μ filter passed through 10 m μ filter.

TABLE 10

SMALL SCALE THERMAL STABILITY TEST DATA FOR TEST BLEND RR-C¹⁴-902

Fuel: RAF-175-63
Labeled Component: Indan-2-C¹⁴

Sp. Act.: 0.02548 $\mu\text{c/ml}$
Total Act.: .12740 $\mu\text{c/5 ml}$

Test No.	Before Storage				After 26 wks. at 130° F			
	1	2	3	4	1	2	3	4
Avg. % T before Testing <u>1/</u>	77.1				57.5			
Final Test Temp., °F	room	394	417	482	room	393	415	488
Avg. % T after Testing <u>1/</u>	77.2	61.5	49.5	34.2	57.5	38.0	34.2	21.0
Δ LT <u>2/</u>		15.6	27.6	42.9		19.5	23.3	36.5
Adherent Deposits, dpm	0	0	25	0	0	10	30	20
μc			0.00001			0.00000	0.00001	0.00001
%			.01				.01	.01
Filterable Deposits, dpm	293	356	687 ^{3/} 3234 ^{4/}	707 ^{3/} 2884 ^{4/}	400 ^{3/} 3244 ^{4/}	421 ^{3/} 2404 ^{4/}	457 ^{3/} 3104 ^{4/}	326 ^{3/} 2504 ^{4/}
μc	0.00013	0.00016	.00031 .00014	0.00032 .00013	0.00018 .00015	.00019 .00011	.00021 .00014	.00015 .00011
%	.10	.12	.24 .11	.25 .10	.14 .11	.15 .08	.16 .11	.08 .09
Total Deposits, μc	.00013	.00016	.00046	.00045	.00033	.00030	.00036	.00027
%	.10	.12	.36	.35	.25	.23	.28	.18

1/ Light transmission at 350 m μ .

2/ Light transmittance loss.

3/ Required prefiltering through 0.45 micron filter.

4/ Filtrate from 0.45 μ filter passed through 10 m μ filter.

TABLE 11

SMALL SCALE THERMAL STABILITY TEST DATA FOR TEST BLEND RR-C¹⁴-903

Fuel: RAF-174-63
Labeled Component: Indan-2-C¹⁴

Sp. Act.: 0.02641 $\mu\text{c/ml}$
Total Act.: .13205 $\mu\text{c/5 ml}$

Test No.	Before Storage				After 26 wks. at 130° F			
	1	2	3	4	1	2	3	4
Avg. % T before Testing <u>1/</u>	95.7				72.1			
Final Test Temp., °F	room	383	389	413	room	385	394	407
Avg. % T after Testing <u>1/</u>	95.8	83.8	81.0	28.0	71.0	40.2	29.0	17.2
Δ LT <u>2/</u>		11.9	14.7	67.7	1.1	31.9	43.1	54.9
Adherent Deposits, dpm	15	15	0	15	0	10	10	30
μc	0.00001	0.00001		0.00001		0.00000	0.00000	0.00001
%	.00	.00		.00				.01
Filterable Deposits, dpm	340	330	162	345	360 ^{3/} 188 ^{4/}	399 ^{3/} 158 ^{4/}	326 ^{3/} 210 ^{4/}	394 ^{3/} 172 ^{4/}
μc	.00015	.00015	0.00007	.00016	0.00016	.00018	.00015	.00018
%	.12	.11	.06	.12	.00008 .12	.00007 .14	.00010 .11	.00008 .14
Total Deposits, μc	.00016	.00016	.00007	.00017	.06 .00024	.05 .00025	.07 .00025	.06 .00027
%	.12	.12	.06	.12	.18	.19	.18	.21

1/ Light transmission at 350 $m\mu$.

2/ Light transmittance loss.

3/ Required prefiltering through 0.45 micron filter.

4/ Filtrate from 0.45 μ filter passed through 10 $m\mu$ filter.

TABLE 12

SMALL SCALE THERMAL STABILITY TEST DATA FOR TEST BLEND RR-C¹⁴-904

Fuel: RAF-159-60X
Labeled Component: Indan-2-C¹⁴

Sp. Act.: 0.02764 $\mu\text{c/ml}$
Total Act.: .13820 $\mu\text{c/5 ml}$

Test No.	Before Storage				After 26 wks. at 130° F			
	1	2	3	4	1	2	3	4
Avg. % T before Testing <u>1/</u>	88.8				90.5			
Final Test Temp., °F	room	416	514	615	room	421	516	610
Avg. % T after Testing <u>1/</u>	88.8	72.2	64.8	59.0	90.5	75.2	65.2	60.0
Δ LT <u>2/</u>		16.6	24.0	29.8		15.3	25.3	30.5
Adherent Deposits, dpm	0	20	35	10	0	25	30	10
μc		0.00001	0.00002	0.00000		0.00001	0.00001	0.00000
%		.00	.01			.01	.01	
Filterable Deposits, dpm	477	694	860	720	301 ^{3/} 797 ^{4/}	349 ^{3/} 880 ^{4/}	538 ^{3/} 680 ^{4/}	617 ^{3/} 875 ^{4/}
μc	0.00022	.00031	.00038	.00032	0.00014	.00016	.00024	.00028
%	.16	.23	.28	.23	.00036 .10	.00040 .12	.00031 .18	.00039 .20
Total Deposits, μc	.00022	.00032	.00040	.00032	.26 .00050	.29 .00057	.23 .00056	.29 .00067
%	.16	.23	.29	.23	.36	.42	.42	.49

1/ Light transmission at 350 m μ .

2/ Light transmittance loss.

3/ Required prefiltering through 0.45 micron filter.

4/ Filtrate from 0.45 μ filter passed through 10 m μ filter.

TABLE 13

SMALL SCALE THERMAL STABILITY TEST DATA FOR TEST BLEND RR-C¹⁴-905

Fuel: SF6-6311
Labeled Component: Indan-2-C¹⁴

Sp. Act.: 0.02586 $\mu\text{c/ml}$
Total Act.: .12930 $\mu\text{c/5 ml}$

Test No.	Before Storage				After 26 wks. at 130° F			
	1	2	3	4	1	2	3	4
Avg. % T before Testing <u>1/</u>	99.7				86.5			
Final Test Temp., °F	room	393	431	503	room	394	428	506
Avg. % T after Testing <u>1/</u>	99.8	83.0	75.0	60.8	86.5	36.8	26.0	7.5
Δ LT <u>2/</u>		16.7	24.7	38.9		49.7	60.5	79.0
Adherent Deposits, dpm	0	20	25	5	10	35	20	40
μc		0.00001	0.00001	0.00000	0.00000	0.00002	0.00001	0.00002
%		.00	.01			.01	.00	.01
Filterable Deposits, dpm	347	549	513	466	$\frac{665^3/}{9594/}$	$\frac{551^3/}{7234/}$	$\frac{536^3/}{8504/}$	$\frac{493^3/}{9234/}$
μc	0.00016	.00025	.00023	.00021	.00030	.00025	.00024	.00022
%	.12	.19	.18	.16	.00043	.00033	.00038	.00042
Total Deposits, μc					.22	.19	.18	.17
%					.32	.24	.29	.31
Total Deposits, μc	.00016	.00026	.00024	.00021	.00073	.00060	.00063	.00066
%	.12	.19	.19	.16	.54	.44	.47	.49

1/ Light transmission at 350 $m\mu$.

2/ Light transmittance loss.

3/ Required prefiltering through 0.45 micron filter.

4/ Filtrate from 0.45 μ filter passed through 10 $m\mu$ filter.

TABLE 14

SMALL SCALE THERMAL STABILITY TEST DATA FOR TEST BLEND RR-C¹⁴-906

Fuel: BJ-64-10-G71
Labeled Component: Indan-2-C¹⁴

Sp. Act.: 0.02678 $\mu\text{c}/\text{ml}$
Total Act.: .13390 $\mu\text{c}/5 \text{ ml}$

Test No.	Before Storage				After 26 wks. at 130° F			
	1	2	3	4	1	2	3	4
Avg. % T before Testing <u>1/</u>	92.8				93.8			
Final Test Temp., °F	room	384	402	445	room	383	401	445
Avg. % T after Testing <u>1/</u>	93.2	71.8	65.2	51.8	93.2	79.2	70.0	53.8
Δ LT <u>2/</u>		21.0	27.6	41.0		14.6	23.8	40.0
Adherent Deposits, dpm	10	30	25	0	5	25	10	45
μc	0.00000	0.00001	0.00001		0.00000	0.00001	0.00000	0.00002
%		.01	.01			.01		.02
Filterable Deposits, dpm	628	750	1015	852	456 ^{3/} 801 ^{4/}	358 ^{3/} 671 ^{4/}	490 ^{3/} 735 ^{4/}	486 ^{3/} 863 ^{4/}
μc	.00028	.00034	.00046	0.00038	.00020	.00016	.00022	.00022
%	.21	.25	.34	.29	.00036 .16 .27	.00030 .12 .23	.00033 .17 .25	.00039 .17 .30
Total Deposits, μc	.00028	.00035	.00047	.00038	.00056	.00047	.00055	.00063
%	.21	.26	.35	.29	.43	.36	.42	.49

1/ Light transmission at 350 $m\mu$.

2/ Light transmittance loss.

3/ Required prefiltering through 0.45 micron filter.

4/ Filtrate from 0.45 μ filter passed through 10 $m\mu$ filter.

TABLE 15

SMALL SCALE THERMAL STABILITY TEST DATA FOR TEST BLEND RR-C¹⁴-907

Fuel: BJ-64-10-G166 Sp. Act.: 0.02600 $\mu\text{c}/\text{ml}$
 Labeled Component: Indan-2-C¹⁴ Total Act.: .13000 $\mu\text{c}/5 \text{ ml}$

Test No.	Before Storage				After 26 wks. at 130° F			
	1	2	3	4	1	2	3	4
Avg. % T before Testing <u>1/</u>	98.6				71.8			
Final Test Temp., °F	room	384	420	488	room	386	418	489
Avg. % T after Testing <u>1/</u>	98.8	76.8	66.8	51.0	71.5	21.8	14.2	6.2
Δ LT <u>2/</u>		21.8	31.8	47.6		50.0	57.6	65.6
Adherent Deposits, dpm	0	25	10	15	0	20	35	10
μc		0.00001	0.00000	0.00001		0.00001	0.00002	0.00000
%		.01		.00		.00	.01	
Filterable Deposits, dpm	367	420	351	511	397 ^{3/} 538 ^{4/}	432 ^{3/} 567 ^{4/}	457 ^{3/} 464 ^{4/}	562 ^{3/} 372 ^{4/}
μc	0.00017	.00019	.00016	.00023	0.00018 .00024	.00020 .00026	.00021 .00021	.00025 .00017
%	.13	.14	.12	.18	.14 .19	.15 .20	.16 .16	.20 .13
Total Deposits, μc	.00017	.00020	.00016	.00024	.00042	.00047	.00043	.00042
%	.13	.15	.12	.18	.33	.35	.33	.33

- 1/ Light transmission at 350 $\text{m}\mu$.
- 2/ Light transmittance loss.
- 3/ Required prefiltering through 0.45 micron filter.
- 4/ Filtrate from 0.45 μ filter passed through 10 $\text{m}\mu$ filter.

TABLE 16

SMALL SCALE THERMAL STABILITY TEST DATA FOR TEST BLEND RR-C¹⁴-909

Fuel: F-63-18
Labeled Component: Indan-2-C¹⁴

Sp. Act.: 0.02664 $\mu\text{c/ml}$
Total Act.: .13320 $\mu\text{c/5 ml}$

Test No.	Before Storage				After 26 wks. at 130° F			
	1	2	3	4	1	2	3	4
Avg. % T before Testing <u>1/</u>	90.9				92.1			
Final Test Temp., °F	room	381	436	541	room	381	440	541
Avg. % T after Testing <u>1/</u>	91.2	79.2	65.2	52.2	92.2	77.8	64.2	50.5
Δ LT <u>2/</u>		11.7	25.7	38.7		14.3	27.9	41.6
Adherent Deposits, dpm	0	15	35	35	0	15	50	85
μc		0.00001	0.00002	0.00002		0.00001	0.00002	0.00004
%		.00	.01	.01		.00	.02	.03
Filterable Deposits, dpm	484	718	840 ^{3/} 585 ^{4/}	718 ^{3/} 532 ^{4/}	393 ^{3/} 407 ^{4/}	380 ^{3/} 564 ^{4/}	544 ^{3/} 521 ^{4/}	669 ^{3/} 496 ^{4/}
μc	0.00022	.00032	.00038 .00026	.00032 .00024	0.00018 .00018	.00017 .00025	.00024 .00024	.00030 .00022
%	.16	.24	.28 .20	.24 .18	.13 .14	.13 .19	.18 .18	.23 .17
Total Deposits, μc	.00022	.00033	.00066	.00058	.00036	.00043	.00050	.00056
%	.16	.24	.49	.43	.27	.32	.38	.43

1/ Light transmission at 350 m μ .

2/ Light transmittance loss.

3/ Required prefiltering through 0.45 micron filter.

4/ Filtrate from 0.45 μ filter passed through 10 m μ filter.

TABLE 17

SMALL SCALE THERMAL STABILITY TEST DATA FOR TEST BLEND RR-C¹⁴-910

Fuel: BJ-64-10-G107 Sp. Act.: 0.02700 $\mu\text{c/ml}$
 Labeled Component: Indan-2-C¹⁴ Total Act.: .13500 $\mu\text{c/5 ml}$

Test No.	Before Storage				After 26 wks. at 130° F			
	1	2	3	4	1	2	3	4
Avg. % T before Testing <u>1/</u>	96.0				97.0			
Final Test Temp., °F	room	399	429	498	room	404	428	496
Avg. % T after Testing <u>1/</u>	95.5	74.8	68.2	58.2	97.2	81.0	73.5	61.8
Δ LT <u>2/</u>		21.2	27.8	37.8		16.0	23.5	35.2
Adherent Deposits, dpm	0	40	45	30	0	35	40	30
μc		0.00002	0.00002	0.00001		0.00002	0.00002	0.00001
%		.01	.01	.01		.01	.01	.01
Filterable Deposits, dpm	598	772	853	723	453 ^{3/} 658 ^{4/}	517 ^{3/} 513 ^{4/}	503 ^{3/} 642 ^{4/}	343 ^{3/} 653 ^{4/}
μc	0.00027	.00035	.00038	.00033	0.00020 .00030	.00023 .00023	.00023 .00029	.00015 .00029
%	.20	.26	.28	.24	.15 .22	.17 .17	.17 .21	.11 .22
Total Deposits, μc	.00027	.00037	.00040	.00034	.00050	.00048	.00054	.00045
%	.20	.27	.29	.25	.37	.35	.39	.34

1/ Light transmission at 350 m μ .

2/ Light transmittance loss.

3/ Required prefiltering through 0.45 micron filter.

4/ Filtrate from 0.45 μ filter passed through 10 m μ filter.

TABLE 18

SMALL SCALE THERMAL STABILITY TEST DATA FOR TEST BLEND RR-C¹⁴-911

Fuel: RAF-178-64 Sp. Act.: 0.02655 $\mu\text{c}/\text{ml}$
 Labeled Component: Indan-2-C¹⁴ Total Act.: .13275 $\mu\text{c}/5 \text{ ml}$

Test No.	Before Storage				After 26 wks. at 130° F			
	1	2	3	4	1	2	3	4
Avg. % T before Testing <u>1/</u>	53.6				47.2			
Final Test Temp., °F	room	362	383	412	room	363	378	405
Avg. % T after Testing <u>1/</u>	54.0	36.2	30.8	11.0	47.0	21.0	18.5	10.5
Δ LT <u>2/</u>		17.4	22.8	42.6		26.2	28.7	36.7
Adherent Deposits, dpm	0	0	10	30	0	20	25	150
μc			0.00000	0.00001		0.00001	0.00001	0.00007
%				.01		.01	.01	.05
Filterable Deposits, dpm	366	419 ^{3/} 344 ^{4/}	452 ^{3/} 269 ^{4/}	511 ^{3/} 220 ^{4/}	253 ^{3/} 243 ^{4/}	500 ^{3/} 319 ^{4/}	708 ^{3/} 238 ^{4/}	525 ^{3/} 221 ^{4/}
μc	0.00017	0.00018 .00016	.00020 .00012	.00023 .00010	0.00011 .00011	.00022 .00014	.00032 .00011	.00024 .00010
%	.12	.14 .12	.15 .09	.17 .07	.08 .08	.17 .11	.24 .08	.18 .07
Total Deposits, μc	.00017	.00034	.00032	.00034	.00022	.00037	.00044	.00041
%	.12	.26	.24	.25	.16	.29	.33	.30

- 1/ Light transmission at 350 $m\mu$.
- 2/ Light transmittance loss.
- 3/ Required prefiltering through 0.45 micron filter.
- 4/ Filtrate from 0.45 μ filter passed through 10 $m\mu$ filter.

TABLE 19

SMALL SCALE THERMAL STABILITY TEST DATA FOR TEST BLEND RR-C¹⁴-925

Fuel: RAF-165-60 Sp. Act.: 0.01514 $\mu\text{c/ml}$
 Labeled Component: n-Hendecane-1-C¹⁴ Total Act.: .07570 $\mu\text{c/5 ml}$

Test No.	Before Storage				After 26 wks. at 130° F			
	1	2	3	4	1	2	3	4
Avg. % T before Testing <u>1/</u>	53.2				53.1			
Final Test Temp., °F	room	394	478	616	room	403	478	619
Avg. % T after Testing <u>1/</u>	52.8	37.8	24.8	19.2	53.0	40.2	26.8	18.5
Δ LT <u>2/</u>		15.4	28.4	34.0		12.9	26.3	34.6
Adherent Deposits, dpm	0	0	0	0	0	0	15	15
μc							0.00001	0.00001
%							.01	.01
Filterable Deposits, dpm	143	135	114	156	384 ^{3/} _{100^{4/}}	480 ^{3/} _{108^{4/}}	417 ^{3/} _{105^{4/}}	348 ^{3/} _{155^{4/}}
μc	0.00006	0.00006	0.00005	0.00007	0.00017	0.00022	.00019	.00016
%	.08	.08	.07	.09	.00004	.00005	.00005	.00007
Total Deposits, μc	.00006	.00006	.00005	.00007	.00021	.00027	.00025	.00024
%	.08	.08	.07	.09	.29	.35	.32	.31

1/ Light transmission at 350 m μ .

2/ Light transmittance loss.

3/ Required prefiltering through 0.45 micron filter.

4/ Filtrate from 0.45 μ filter passed through 10 m μ filter.

TABLE 20

SMALL SCALE THERMAL STABILITY TEST DATA FOR TEST BLEND RR-C¹⁴-926

Fuel: RAF-174-63 Sp. Act.: 0.03146 $\mu\text{c}/\text{ml}$
 Labeled Component: n-Hendecane-1-C¹⁴ Total Act.: .15730 $\mu\text{c}/5 \text{ ml}$

Test No.	Before Storage				After 26 wks. at 130° F			
	1	2	3	4	1	2	3	4
Avg. % T before Testing <u>1/</u>	96.1				68.7			
Final Test Temp., °F	room	384	394	409	room	381	392	411
Avg. % T after Testing <u>1/</u>	96.2	83.0	73.0	26.8	67.2	39.5	27.2	15.0
Δ LT <u>2/</u>		13.1	23.1	69.3	1.5	29.2	41.5	53.7
Adherent Deposits, dpm	10	25	0	0	0	0	0	15
μc	0.00000	0.00001						0.00001
%		.01						.00
Filterable Deposits, dpm	318	216	288	233	413 ^{3/} 170 ^{4/}	681 ^{3/} 196 ^{4/}	741 ^{3/} 336 ^{4/}	720 ^{3/} 113 ^{4/}
μc	.00014	.00010	0.00013	0.00011	0.00019 .00008	0.00031 .00009	0.00033 .00015	.00032 .00005
%	.09	.06	.08	.07	.12 .05	.20 .06	.21 .10	.21 .03
Total Deposits, μc	.00014	.00011	.00013	.00011	.00027	.00040	.00048	.00038
%	.09	.07	.08	.07	.17	.26	.31	.24

1/ Light transmission at 350 m μ .

2/ Light transmittance loss.

3/ Required prefiltering through 0.45 micron filter.

4/ Filtrate from 0.45 μ filter passed through 10 m μ filter.

TABLE 21

SMALL SCALE THERMAL STABILITY TEST DATA FOR TEST BLEND RR-C¹⁴-927

Fuel: BJ-64-10-G107 Sp. Act.: 0.02706 $\mu\text{c/ml}$
 Labeled Component: n-Hendecane-1-C¹⁴ Total Act.: .13530 $\mu\text{c/5 ml}$

Test No.	Before Storage				After 26 wks. at 130° F			
	1	2	3	4	1	2	3	4
Avg. % T before Testing <u>1/</u>	96.3				96.2			
Final Test Temp., °F	room	400	437	490	room	403	430	495
Avg. % T after Testing <u>1/</u>	96.2	79.0	72.5	61.2	96.2	80.2	74.2	60.8
Δ LT <u>2/</u>		17.3	23.8	35.1		16.0	22.0	35.4
Adherent Deposits, dpm	0	0	0	0	0	0	0	80
μc								0.00004
%								.03
Filterable Deposits, dpm	297	301	305	275	$\frac{7893}{2214}$	$\frac{5913}{994}$	$\frac{6933}{1394}$	$\frac{8853}{1334}$
μc	0.00013	0.00014	0.00014	0.00012	0.00036	0.00027	0.00031	.00040
%	.10	.10	.10	.09	.00010	.00004	.00006	.00006
					.26	.20	.23	.29
					.07	.03	.04	.04
Total Deposits, μc	.00013	.00014	.00014	.00012	.00046	.00031	.00037	.00050
%	.10	.10	.10	.09	.33	.23	.27	.36

1/ Light transmission at 350 $m\mu$.

2/ Light transmittance loss.

3/ Required prefiltering through 0.45 micron filter.

4/ Filtrate from 0.45 μ filter passed through 10 $m\mu$ filter.

TABLE 22

SMALL SCALE THERMAL STABILITY TEST DATA FOR TEST BLEND RR-C¹⁴-928

Fuel: BJ-64-10-G166 Sp. Act.: 0.03035 $\mu\text{c/ml}$
 Labeled Component: n-Hendecane-1-C¹⁴ Total Act.: .15175 $\mu\text{c/5 ml}$

Test No.	Before Storage				After 26 wks. at 130° F			
	1	2	3	4	1	2	3	4
Avg. % T before Testing <u>1/</u>	98.7				65.2			
Final Test Temp., °F	room	382	420	491	room	383	419	485
Avg. % T after Testing <u>1/</u>	99.2	80.2	69.2	46.5	64.5	28.0	14.0	6.2
Δ LT <u>2/</u>		18.5	29.5	52.2		37.2	51.2	59.0
Adherent Deposits, dpm	0	15	0	0	10	0	0	15
μc		0.00001			0.00000			0.00001
%		.00						.00
Filterable Deposits, dpm	297	428	373	68	677 ^{3/} _{1924/}	711 ^{3/} _{1104/}	568 ^{3/} _{1274/}	761 ^{3/} _{1694/}
μc	0.00013	.00019	0.00017	0.00003	.00030	0.00032	0.00026	.00034
%	.09	.13	.11	.02	.00009	.00005	.00006	.00008
Total Deposits, μc	.06	.03	.04	.05	.20	.21	.17	.23
%	.09	.13	.11	.02	.26	.24	.21	.28

1/ Light transmission at 350 $m\mu$.

2/ Light transmittance loss.

3/ Required prefiltering through 0.45 micron filter.

4/ Filtrate from 0.45 μ filter passed through 10 $m\mu$ filter.

TABLE 23

SMALL SCALE THERMAL STABILITY TEST DATA FOR TEST BLEND RR-C¹⁴-929

Fuel: RAF-175-63 Sp. Act.: 0.02807 $\mu\text{c}/\text{ml}$
 Labeled Component: n-Hendecane-1-C¹⁴ Total Act.: .14035 $\mu\text{c}/5 \text{ ml}$

Test No.	Before Storage				After 26 wks. at 130° F			
	1	2	3	4	1	2	3	4
Avg. % T before Testing <u>1/</u>	77.4				61.8			
Final Test Temp., °F	room	388	415	490	room	390	414	483
Avg. % T after Testing <u>1/</u>	77.8	65.5	47.2	35.2	61.5	41.8	35.2	23.5
Δ LT <u>2/</u>		11.9	30.2	42.2		20.0	26.6	38.3
Adherent Deposits, dpm	0	0	10	0	0	0	0	0
μc			0.00000					
%								
Filterable Deposits, dpm	228	177	890 ^{3/}	776 ^{3/}	613 ^{3/}	654 ^{3/}	701 ^{3/}	602 ^{3/}
μc	0.00010	0.00008	3294 ^{4/} .00040	2744 ^{4/} 0.00035	554 ^{4/} 0.00028	1694 ^{4/} 0.00030	2704 ^{4/} 0.00032	2574 ^{4/} 0.00027
%	.07	.06	.00015 .28	.00012 .25	.00002 .19	.00008 .21	.00012 .22	.00012 .19
Total Deposits, μc	.00010	.00008	.10 .00055	.09 .00047	.02 .00030	.05 .00038	.08 .00044	.08 .00039
%	.07	.06	.38	.34	.21	.26	.30	.27

1/ Light transmission at 350 $m\mu$.

2/ Light transmittance loss.

3/ Required prefiltering through 0.45 micron filter.

4/ Filtrate from 0.45 μ filter passed through 10 $m\mu$ filter.

TABLE 24

SMALL SCALE THERMAL STABILITY TEST DATA FOR TEST BLEND RR-C¹⁴-930

Fuel: RAF-159-60X Sp. Act.: 0.03017 $\mu\text{c/ml}$
 Labeled Component: n-Hendecane-1-C¹⁴ Total Act.: .15085 $\mu\text{c/5 ml}$

Test No.	Before Storage				After 26 wks. at 130° F			
	1	2	3	4	1	2	3	4
Avg. % T before Testing <u>1/</u>	89.3				91.0			
Final Test Temp., °F	room	427	527	622	room	421	524	615
Avg. % T after Testing <u>1/</u>	89.8	68.5	59.5	58.0	91.0	76.2	64.8	55.0
Δ LT <u>2/</u>		20.8	29.8	31.3		14.8	26.2	36.0
Adherent Deposits, dpm	0	0	15	10	15	0	0	15
μc			0.00001	0.00000	0.00001			0.00001
%			.00		.00			.00
Filterable Deposits, dpm	238	385	209	234	6713/ 118 ^{4/}	8473/ 187 ^{4/}	7813/ 253 ^{4/}	10823/ 264 ^{4/}
μc	0.00011	0.00017	.00009	.00011	.00030	0.00038	0.00035	.00049
%	.07	.11	.06	.07	.00005	.00008	.00011	.00012
Total Deposits, μc	.00011	.00017	.00010	.00011	.04	.06	.08	.08
%	.07	.11	.06	.07	.20	.26	.24	.32
					.04	.06	.08	.08
Total Deposits, %	.07	.11	.06	.07	.24	.32	.32	.40

1/ Light transmission at 350 $m\mu$.

2/ Light transmittance loss.

3/ Required prefiltering through 0.45 micron filter.

4/ Filtrate from 0.45 μ filter passed through 10 $m\mu$ filter.

TABLE 25

SMALL SCALE THERMAL STABILITY TEST DATA FOR TEST BLEND RR-C¹⁴-931

Fuel: BJ-64-10-G71 Sp. Act.: 0.02912 $\mu\text{c/ml}$
 Labeled Component: n-Hendecane-1-C¹⁴ Total Act.: .14560 $\mu\text{c/5 ml}$

Test No.	Before Storage				After 26 wks. at 130° F			
	1	2	3	4	1	2	3	4
Avg. % T before Testing <u>1/</u>	93.4				93.6			
Final Test Temp., °F	room	382	399	448	room	388	405	442
Avg. % T after Testing <u>1/</u>	93.8	71.0	67.0	50.8	93.0	71.5	70.0	53.0
Δ LT <u>2/</u>		22.4	26.4	42.6		22.1	23.6	40.6
Adherent Deposits, dpm	0	10	0	0	0	0	0	0
μc		0.00000						
%								
Filterable Deposits, dpm	175	200	162	167	827 ^{3/} 458 ^{4/}	729 ^{3/} 479 ^{4/}	553 ^{3/} 444 ^{4/}	956 ^{3/} 389 ^{4/}
μc	0.00008	.00009	0.00007	0.00008	0.00037 .00021	0.00033 .00022	0.00025 .00020	0.00043 .00018
%	.05	.06	.05	.05	.26 .14	.23 .15	.17 .14	.30 .12
Total Deposits, μc	.00008	.00009	.00007	.00008	.00058	.00055	.00045	.00061
%	.05	.06	.05	.05	.40	.38	.31	.42

1/ Light transmission at 350 m μ .

2/ Light transmittance loss.

3/ Required prefiltering through 0.45 micron filter.

4/ Filtrate from 0.45 μ filter passed through 10 m μ filter.

TABLE 26

SMALL SCALE THERMAL STABILITY TEST DATA FOR TEST BLEND RR-C¹⁴-932

Fuel: BJ-63-10-B75 Sp. Act.: 0.02754 $\mu\text{c}/\text{ml}$
 Labeled Component: n-Hendecane-1-C¹⁴ Total Act.: .13770 $\mu\text{c}/5 \text{ ml}$

Test No.	Before Storage				After 26 wks. at 130° F			
	1	2	3	4	1	2	3	4
Avg. % T before Testing <u>1/</u>	60.2				49.3			
Final Test Temp., °F	room	402	501	623	room	402	506	621
Avg. % T after Testing <u>1/</u>	60.5	43.0	38.2	32.2	49.2	35.2	20.2	12.5
Δ LT <u>2/</u>		17.2	22.0	28.0		14.1	29.1	36.8
Adherent Deposits, dpm	0	0	0	20	0	0	0	0
μc				0.00001				
%				.00				
Filterable Deposits, dpm	163	151	134	201	660 ^{3/} / _{449^{4/}}	761 ^{3/} / _{479^{4/}}	771 ^{3/} / _{300^{4/}}	504 ^{3/} / _{378^{4/}}
μc	0.00007	0.00007	0.00006	.00009	0.00030	0.00034	0.00035	0.00023
%	.05	.05	.04	.06	.00020 .22	.00022 .25	.00014 .25	.00017 .16
Total Deposits, μc	.00007	.00007	.00006	.00010	.00050	.00056	.00049	.00040
%	.05	.05	.04	.06	.37	.41	.35	.28

1/ Light transmission at 350 $m\mu$.

2/ Light transmittance loss.

3/ Required prefiltering through 0.45 micron filter.

4/ Filtrate from 0.45 μ filter passed through 10 $m\mu$ filter.

TABLE 27

SMALL SCALE THERMAL STABILITY TEST DATA FOR TEST BLEND RR-C¹⁴-933

Fuel: SF6-6311 Sp. Act.: 0.02541 $\mu\text{c/ml}$
 Labeled Component: n-Hendecane-1-C¹⁴ Total Act.: .12705 $\mu\text{c/5 ml}$

Test No.	Before Storage				After 26 wks. at 130° F			
	1	2	3	4	1	2	3	4
Avg. % T before Testing <u>1/</u>	99.8				85.2			
Final Test Temp., °F	room	389	430	502	room	390	433	500
Avg. % T after Testing <u>1/</u>	99.2	86.2	72.5	62.0	86.0	48.8	32.5	10.5
Δ LT <u>2/</u>		13.6	27.3	37.8		36.4	52.7	74.7
Adherent Deposits, dpm	0	0	15	0	0	10	0	0
μc			0.00001			0.00000		
%			.00					
Filterable Deposits, dpm	171	158	242	279	727 ^{3/} 344 ^{4/}	608 ^{3/} 304 ^{4/}	454 ^{3/} 352 ^{4/}	625 ^{3/} 304 ^{4/}
μc	0.00008	0.00007	.00011	0.00013	0.00033	.00027	0.00020	0.00028
%	.06	.06	.09	.10	.26	.21	.16	.22
Total Deposits, μc	.00008	.00007	.00012	.00013	.00049	.00041	.00036	.00042
%	.06	.06	.09	.10	.38	.32	.28	.33

1/ Light transmission at 350 $m\mu$.

2/ Light transmittance loss.

3/ Required prefiltering through 0.45 micron filter.

4/ Filtrate from 0.45 μ filter passed through 10 $m\mu$ filter.

TABLE 28

SMALL SCALE THERMAL STABILITY TEST DATA FOR TEST BLEND RR-C¹⁴- 934

Fuel: F-63-18 Sp. Act.: 0.03009 $\mu\text{c}/\text{ml}$
 Labeled Component: n-Hendecane-1-C¹⁴ Total Act.: .15045 $\mu\text{c}/5 \text{ ml}$

Test No.	Before Storage				After 26 wks. at 130° F			
	1	2	3	4	1	2	3	4
Avg. % T before Testing <u>1/</u>	91.0				92.0			
Final Test Temp., °F	room	385	438	547	room	388	440	539
Avg. % T after Testing <u>1/</u>	91.0	78.0	63.8	51.2	92.0	74.0	65.8	50.8
Δ LT <u>2/</u>		13.0	27.2	39.8		18.0	26.2	41.2
Adherent Deposits, dpm	0	0	10	0	0	0	0	15
μc			0.00000					0.00001
%								.00
Filterable Deposits, dpm	149	178	649 ^{3/} / _{246^{4/}}	692 ^{3/} / _{186^{4/}}	667 ^{3/} / _{331^{4/}}	643 ^{3/} / _{418^{4/}}	583 ^{3/} / _{410^{4/}}	622 ^{3/} / _{375^{4/}}
μc	0.00007	0.00008	.00029	0.00031	0.00030	0.00029	0.00026	.00028
%	.04	.05	.00011 .19 .07	.00008 .20 .06	.00015 .20 .10	.00019 .19 .12	.00019 .18 .12	.00017 .19 .11
Total Deposits, μc	.00007	.00008	.00040	.00039	.00045	.00048	.00045	.00046
%	.04	.05	.26	.26	.30	.31	.30	.30

1/ Light transmission at 350 $m\mu$.

2/ Light transmittance loss.

3/ Required prefiltering through 0.45 micron filter.

4/ Filtrate from 0.45 μ filter passed through 10 $m\mu$ filter.

TABLE 29

SMALL SCALE THERMAL STABILITY TEST DATA FOR TEST BLEND RR-C¹⁴-935

Fuel: BJ-64-10-G74 Sp. Act.: 0.02888 $\mu\text{c}/\text{ml}$
 Labeled Component: n-Hendecane-1-C¹⁴ Total Act.: .14440 $\mu\text{c}/5 \text{ ml}$

Test No.	Before Storage				After 26 wks. at 130° F			
	1	2	3	4	1	2	3	4
Avg. % T before Testing <u>1/</u>	97.1				8.5			
Final Test Temp., °F	room	367	380	397	room	359	379	392
Avg. % T after Testing <u>1/</u>	97.2	90.2	86.8	46.0	8.8	8.2	9.5	8.5
Δ LT <u>2/</u>		6.9	10.3	51.1				
Adherent Deposits, dpm	0	0	10	15	0	0	10	0
μc			0.00000	0.00001			0.00000	
%				.00				
Filterable Deposits, dpm	202	150	97	6243/ 1034/	8793/ 4034/	6173/ 5804/	8023/ 5624/	7543/ 5844/
μc	0.00009	0.00007	.00004	.00028	0.00040	0.00028	.00036	0.00034
%	.06	.05	.03	.19	.28	.20	.26	.24
Total Deposits, μc	.00009	.00007	.00004	.00033	.00058	.00054	.00061	.00060
%	.06	.05	.03	.22	.41	.38	.44	.42

1/ Light transmission at 350 $m\mu$.

2/ Light transmittance loss.

3/ Required prefiltering through 0.45 micron filter.

4/ Filtrate from 0.45 μ filter passed through 10 $m\mu$ filter.

TABLE 30

SMALL SCALE THERMAL STABILITY TEST DATA FOR TEST BLEND RR-C¹⁴-936

Fuel: RAF-178-64 Sp. Act.: 0.02797 $\mu\text{c}/\text{ml}$
 Labeled Component: n-Hendecane-1-C¹⁴ Total Act.: .13985 $\mu\text{c}/5 \text{ ml}$

Test No.	Before Storage				After 26 wks. at 130° F			
	1	2	3	4	1	2	3	4
Avg. % T before Testing <u>1/</u>	54.0				51.5			
Final Test Temp., °F	room	364	380	407	room	367	383	404
Avg. % T after Testing <u>1/</u>	53.8	38.0	33.2	13.0	51.0	24.2	21.2	13.0
Δ LT <u>2/</u>		16.0	20.8	41.0		27.3	30.3	38.5
Adherent Deposits, dpm	0	0	10	15	0	0	0	0
μc			0.00000	0.00001				
%				.00				
Filterable Deposits, dpm	182	9013/ 1524/	7403/ 1154/	4123/ 844/	2183/ 4074/	4433/ 4344/	6443/ 3034/	8093/ 3994/
μc	0.00008	0.00041	.00033	.00019	0.00010	0.00020	0.00029	0.00036
%	.06	.29	.24	.13	.07	.14	.20	.26
Total Deposits, μc	.00008	.00048	.00038	.00023	.00028	.00040	.00043	.00054
%	.06	.34	.28	.16	.20	.28	.30	.38

1/ Light transmission at 350 $m\mu$.

2/ Light transmittance loss.

3/ Required prefiltering through 0.45 micron filter.

4/ Filtrate from 0.45 μ filter passed through 10 $m\mu$ filter.

TABLE 31

RADIOACTIVITY BALANCE FOR THERMALLY STRESSED TEST BLEND RR-C¹⁴-899

Fuel: BJ-63-10-B75

Labeled Component: Indan-2-C¹⁴

Storage: 26 weeks, 130° F

Test No.	Final Temp., °F	Test Blend Radioactivity, $\mu\text{C}/5\text{ ml}$	Adherent Deposits, μC	Filterable Deposits, μC	Total Deposits, μC	Filtrate		Radioactivity Balance	
						Sp. Act., $\mu\text{C}/\text{ml}$	Total Act., $\mu\text{C}/5\text{ ml}$	μC	%
1	room	0.09075	0.00000	0.00059	0.00059	0.01772	0.08860	- 0.00156	- 1.72
2	401	.09075	.00001	.00053	.00054	.01742	.08710	- .00311	- 3.43
3	502	.09075	.00001	.00057	.00058	.01749	.08745	- .00272	- 3.00
4	622	.09075	.00003	.00057	.00060	.01752	.08760	- .00255	- 2.81
									+0.22 ^{1/}

^{1/} Radioactivity change incurred during 26 weeks storage at 130° F.

Contrails

TABLE 32

RADIOACTIVITY BALANCE FOR THERMALLY STRESSED TEST BLEND RR-C¹⁴-900

Fuel: BJ-64-10-G74
 Labeled Component: Indan-2-C¹⁴
 Storage: 26 weeks, 130° F

Test No.	Final Temp., °F	Test Blend Radioactivity, $\mu\text{c}/5\text{ ml}$	Adherent Deposits, μc	Filterable Deposits, μc	Total Deposits, μc	Filtrate		Radioactivity Balance	
						Sp. Act., $\mu\text{c}/\text{ml}$	Total Act., $\mu\text{c}/5\text{ ml}$	μc	%
1	room	0.12520	0.00000	0.00026	0.00026	0.02400	0.12000	-0.00494	-3.94
2	363	.12520	.00000	.00028	.00028	.02418	.12090	-.00402	-3.21
3	377	.12520	.00000	.00025	.00025	.02403	.12015	-.00480	-3.83
4	401	.12520	.00002	.00027	.00029	.02432	.12160	-.00331	-2.64

1/ Radioactivity change incurred during 26 weeks storage at 130° F.

Contrails

TABLE 33

RADIOACTIVITY BALANCE FOR THERMALLY STRESSED TEST BLEND RR-C¹⁴-925

Fuel: RAF-165-60
 Labeled Component: n-Hendecane-1-C¹⁴
 Storage: 26 weeks, 130° F

Test No.	Final Temp., °F	Test Blend Radioactivity, $\mu\text{c}/5\text{ ml}$	Adherent Deposits, μc	Filterable Deposits, μc	Total Deposits, μc	Filtrate		Radioactivity Balance	
						Sp. Act., $\mu\text{c}/\text{ml}$	Total Act., $\mu\text{c}/5\text{ ml}$	μc	%
1	room	0.07570	0.00000	0.00021	0.00021	0.01487	0.07435	-0.00114	-1.50
2	403	.07570	.00000	.00027	.00027	.01475	.07375	-.00168	-2.22
3	478	.07570	.00001	.00024	.00025	.01482	.07410	-.00135	-1.78
4	619	.07570	.00001	.00023	.00024	.01499	.07495	-.00051	-0.67

1/ Radioactivity change incurred during 26 weeks storage at 130° F.

Contrails

TABLE 34

RADIOACTIVITY BALANCE FOR THERMALLY STRESSED TEST BLEND RR-C¹⁴-926

Fuel: RAF-174-63
 Labeled Component: n-Hendecane-1-C¹⁴
 Storage: 26 weeks, 130° F

Test No.	Final Temp., °F	Test Blend Radioactivity, $\mu\text{c}/5\text{ ml}$	Adherent Deposits, μc	Filterable Deposits, μc	Total Deposits, μc	Filtrate		Radioactivity Balance	
						Sp. Act., $\mu\text{c}/\text{ml}$	Total Act., $\mu\text{c}/5\text{ ml}$	μc	%
1	room	0.15730	0.00000	0.00027	0.00027	0.03181	0.15905	+0.00202	+1.28
2	381	.15730	.00000	.00040	.00040	.03132	.15660	-.00030	-0.19
3	392	.15730	.00000	.00048	.00048	.03189	.15945	+.00263	+1.67
4	411	.15730	.00001	.00037	.00038	.03159	.15795	+.00103	+0.65

1/ Radioactivity change incurred during 26 weeks storage at 130° F.

TABLE 35

RADIOACTIVITY BALANCE FOR THERMALLY STRESSED TEST BLEND RR-C¹⁴-927

Fuel: BJ-64-10-G107
 Labeled Component: n-Hendecane-1-C¹⁴
 Storage: 26 weeks, 130° F

Test No.	Final Temp., °F	Test Blend Radioactivity, $\mu\text{C}/5\text{ ml}$	Adherent Deposits, μC	Filterable Deposits, μC	Total Deposits, μC	Filtrate		Radioactivity Balance	
						Sp. Act., $\mu\text{C}/\text{ml}$	Total Act., $\mu\text{C}/5\text{ ml}$	μC	%
1	room	0.13530	0.00000	0.00046	0.00046	0.02736	0.13680	+0.00196	+1.45
2	403	.13530	.00000	.00031	.00031	.02654	.13270	-.00229	-1.69
3	430	.13530	.00000	.00037	.00037	.02701	.13505	+0.00012	+0.09
4	495	.13530	.00004	.00046	.00050	.02664	.13320	-.00160	-1.18

1/ Radioactivity change incurred during 26 weeks storage at 130° F.

TABLE 36

RADIOACTIVITY BALANCE FOR THERMALLY STRESSED TEST BLEND RR-C¹⁴-928

Fuel: BJ-64-10-G166
 Labeled Component: n-Hendecane-1-C¹⁴
 Storage: 26 weeks, 130° F

Test No.	Final Temp., °F	Test Blend Radioactivity, $\mu\text{c}/5\text{ ml}$	Adherent Deposits, μc	Filterable Deposits, μc	Total Deposits, μc	Filtrate		Radioactivity Balance	
						Sp. Act., $\mu\text{c}/\text{ml}$	Total Act., $\mu\text{c}/5\text{ ml}$	μc	%
1	room	0.15175	0.00000	0.00039	0.00039	0.02999	0.14995	-0.00141	-0.93
2	383	.15175	.00000	.00037	.00037	.03029	.15145	+0.00007	+0.05
3	419	.15175	.00000	.00032	.00032	.03025	.15125	-.00018	-0.12
4	485	.15175	.00001	.00042	.00043	.02997	.14985	-.00147	-0.97
1/ Radioactivity change incurred during 26 weeks storage at 130° F.									

TABLE 37

RADIOTRACER CONTRIBUTION TO TOTAL DEPOSITS AT APPROXIMATELY 400° F

Fuel	Before Storage			After Storage		
	Actual Temp.	Total Deposits μc	Total Deposits %	Actual Temp.	Total Deposits μc	Total Deposits %
<u>Hendecane-C¹⁴</u>						
-G71	399	0.00007	0.05	405	0.00045	0.31
-G74	397	.00033	.22	392	.00060	.42
-B75	402	.00007	.05	402	.00056	.41
-G107	400	.00014	.10	403	.00031	.23
SF6-6311	389	.00007	.06	390	.00041	.32
F-63-18	385	.00008	.05	388	.00048	.31
RAF-165-60	394	.00006	.08	403	.00026	.35
RAF-174-63	394	.00013	.08	392	.00048	.31
RAF-175-63	388	.00008	.06	390	.00037	.26
-G166	382	.00020	.13	383	.00037	.24
RAF-159-60X	427	.00017	.11	421	.00047	.32
RAF-178-64	407	.00023	.16	404	.00054	.38
<u>Dodecene-1-C¹⁴</u>						
-G71	386	0.00017	0.13	387	0.00020	0.16
-G74	403	.00004	.09	412	.00012	.23
-B75	401	.00017	.13	402	.00066	.53
-G107	399	.00017	.11	406	.00035	.21
SF6-6311	391	.00019	.12	398	.00068	.42
F-63-18	381	.00020	.13	381	.00049	.31
RAF-165-60	426	.00011	.14	421	.00007	.09
RAF-174-63	404	.00010	.09	401	.00033	.30
RAF-175-63	409	.00008	.05	394	.00050	.31
-G166	383	.00025	.16	387	.00055	.34
RAF-159-60X	427	.00014	.10	422	.00044	.30
RAF-178-64	410	.00047	.29	409	.00044	.28

TABLE 37

RADIOTRACER CONTRIBUTION TO TOTAL DEPOSITS AT APPROXIMATELY 400° F
Continued

Fuel	Before Storage			After Storage		
	Actual Temp.	Total Deposits μc	Total Deposits %	Actual Temp.	Total Deposits μc	Total Deposits %
<u>1,2,3,5-Tetramethylbenzene</u>						
-G71	401	0.00011	0.09	399	0.00030	0.23
-G74	396	.00036	.27	403	.00046	.37
-B75	391	.00013	.09	408	.00047	.35
-G107	401	.00022	.18	407	.00052	.42
SF6-6311	394	.00019	.14	396	.00051	.34
F-63-18	386	.00011	.08	387	.00045	.32
RAF-165-60	399	.00005	.04	405	.00037	.29
RAF-174-63	404	.00006	.04	405	.00039	.29
RAF-175-63	390	.00002	.02	394	.00050	.40
-G166	417	.00013	.09	418	.00044	.31
RAF-159-60X	427	.00014	.11	422	.00046	.37
RAF-178-64	406	.00029	.21	410	.00027	.20
<u>Diphenyl-C¹⁴</u>						
-G71						
-G74	399	0.00025	0.12	390	0.00070	0.36
-B75						
-G107						
SF6-6311						
F-63-18	385	.00040	.21	380	.00065	.33
RAF-165-60	436	.00026	.16	435	.00025	.16
RAF-174-63	397	.00049	.25	397	.00052	.26
RAF-175-63	388	.00064	.32	392	.00069	.34
-G166						
RAF-159-60X						
RAF-178-64						

TABLE 37

RADIOTRACER CONTRIBUTION TO TOTAL DEPOSITS AT APPROXIMATELY 400° F
Continued

Fuel	Before Storage			After Storage		
	Actual Temp.	Total Deposits μc	Total Deposits %	Actual Temp.	Total Deposits μc	Total Deposits %
<u>Indan-2-C¹⁴</u>						
-G71	402	0.00047	0.35	401	0.00055	0.42
-G74	396	.00045	.37	401	.00028	.23
-B75	393	.00036	.39	401	.00054	.59
-G107	399	.00037	.27	404	.00048	.35
SF6-6311	393	.00026	.20	394	.00059	.44
F-63-18	381	.00033	.25	381	.00043	.32
RAF-165-60	402	.00018	.14	402	.00035	.27
RAF-174-63	413	.00017	.12	407	.00027	.21
RAF-175-63	394	.00016	.12	393	.00030	.23
-G166	384	.00020	.15	386	.00046	.35
RAF-159-60X	416	.00032	.23	421	.00056	.42
RAF-178-64	412	.00034	.25	405	.00040	.30
<u>Tetralin-C¹⁴</u>						
-G71	387	0.00042	0.14	387	0.00059	0.19
-G74	408	.00049	.18	404	.00078	.28
-B75	395	.00054	.16	406	.00210	.65
-G107	401	.00041	.12	404	.00105	.31
SF6-6311	389	.00023	.11	397	.00212	.94
F-63-18	384	.00038	.12	383	.00106	.33
RAF-165-60	422	.00018	.06	422	.00046	.15
RAF-174-63	398	.00043	.18	394	.00092	.31
RAF-175-63	393	.00020	.07	395	.00061	.21
-G166	389	.00037	.11	389	.00191	.60
RAF-159-60X	433	.00052	.17	422	.00108	.35
RAF-178-64	404	.00108	.31	403	.00143	.40

TABLE 37

RADIOTRACER CONTRIBUTION TO TOTAL DEPOSITS AT APPROXIMATELY 400° F
Continued

Fuel	Before Storage			After Storage		
	Actual Temp.	Total Deposits μc	Total Deposits %	Actual Temp.	Total Deposits μc	Total Deposits %
<u>2-Methyl-C¹⁴-Naphthalene</u>						
-G71	399	0.00036	0.19	400	0.00095	0.49
-G74	394	.00065	.39	396	.00075	.44
-B75	402	.00064	.34	408	.00110	.61
-G107	400	.00044	.25	406	.00091	.52
SF6-6311	383	.00040	.22	391	.00068	.36
F-63-18	381	.00045	.26	382	.00080	.46
RAF-165-60	424	.00028	.15	429	.00087	.47
RAF-174-63	399	.00037	.21	405	.00050	.27
RAF-175-63	386	.00020	.11	393	.00062	.32
-G166	415	.00025	.14	423	.00063	.35
RAF-159-60X	415	.00040	.22	420	.00108	.59
RAF-178-64	404	.00046	.26	402	.00067	.37
<u>Di-t-butyl-C¹⁴-p-cresol</u>						
-G71						
-G74	395	0.00128	0.61	393	0.00559	2.67
-B75	398	.00206	.59	404	.00547	1.57
-G107						
SF6-6311						
F-63-18	384	.00340	.83	380	.00287	.69
RAF-165-60	439	.00051	.48	437	.00060	.55
RAF-174-63	390	.00196	.50	400	.00515	1.33
RAF-175-63	389	.00249	.68	390	.00407	1.11
-G166	383	.00046	.59	383	.00058	.77
RAF-159-60X	452	.00081	.47	451	.00170	1.00
RAF-178-64	399	.00734	1.90	408	.00907	2.36

TABLE 38

LOSS OF RADIOACTIVITY FROM BLENDS CONTAINING
2,6-di-t-BUTYL-C¹⁴-p-CRESOL AS THE RADIOTRACER

Blend No.	Radiotracer, ppm	Specific Activity, $\mu\text{c/ml}$		Radioactivity Loss		Weight Loss mg/ml
		Before Test	After Test	$\mu\text{c/ml}$	%	
<u>Fuel RAF-165-60</u>						
993	4200	1.0540	1.0036	0.0504	4.78	0.1707
993-1	2100	.5242	.4895	.0347	6.62	.1176
993-2	1000	.2601	.2343	.0258	9.89	.0871
993-3	420	.1039	.0890	.0149	14.39	.0506
993-4	100	.02599	.02060	.00539	20.74	.0182
993-5	10	.002658	.002172	.000486	18.28	.0016
<u>Fuel RAF-175-63</u>						
983	4250	1.0508	1.0091	0.0417	3.97	0.1411
987	2200	.5230	.4802	.0428	8.18	.1448
988	1000	.2539	.2189	.0350	13.78	.1184
989	450	.1061	.0843	.0218	20.52	.0736
976	10	.01960	.01398	.00562	28.68	.0190
<u>Fuel JP-5</u>						
999	4200	1.0448	1.0282	0.0166	1.60	0.0578
999-1	2100	.5189	.5025	.0164	3.16	.0578
999-2	1000	.2558	.2391	.0167	6.54	.0578
999-3	420	.1041	.0923	.0118	11.36	.0408
999-4	100	.02544	.02054	.00490	19.26	.0167
999-5	20	.005320	.004145	.001175	22.09	.0040
997	23	.04524	.03474	.01050	23.21	.0043

APPENDIX IV

PROCEDURES FOR ORGANIC SYNTHESIS
OF ALKYLATED INDENES AND INDANS

APPENDIX IV

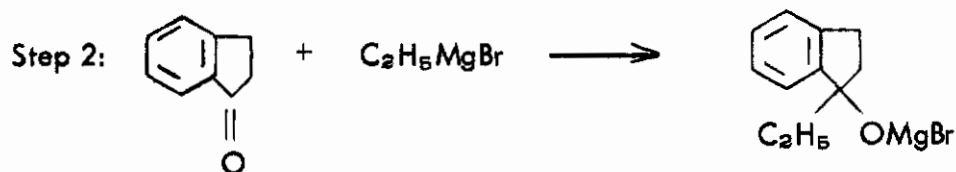
SYNTHESIS OF 3-METHYLINDENE AND 3-ETHYLINDENE

The principal steps of this synthesis include a Grignard reaction of an appropriate alkyl ketone. The product is hydrolyzed to obtain a corresponding indanol, followed by a dehydration step with phosphoric acid to the methyl or ethylindene. The procedure for 3-ethylindene is described in detail below.



The glassware for the Grignard reaction was dried at 110° C and cooled in a desiccator. The magnesium turnings (0.026 g atom, plus a small excess) were washed in ether and dried at 110° C. After it was cooled, the magnesium was placed in a 50-ml two-necked flask, and a magnetic stirrer bar was put into the flask. A 25-ml addition funnel was mounted on the center neck of the flask, and a reflux condenser was mounted on the side neck.

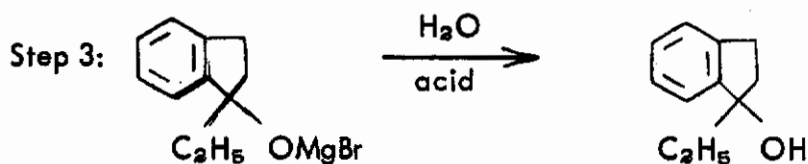
Two milliliters (0.026 mole) of ethyl bromide was measured into the addition funnel. Two milliliters of anhydrous ethyl ether was introduced into the flask through the condenser. The magnetic stirrer was turned on, and several drops of ethyl bromide were added from the addition funnel to the ether and magnesium in the flask. As soon as the reaction had started, as indicated by a cloudiness in the solution, an additional 8 ml of ether was added through the condenser and the remainder of the ethyl bromide was added dropwise. An ice-water bath was used to cool the flask while the ethyl bromide was being added. After all the ethyl bromide had been added and the reaction had subsided, the water bath was heated to about 40° C and the reaction mixture refluxed for approximately 30 minutes.



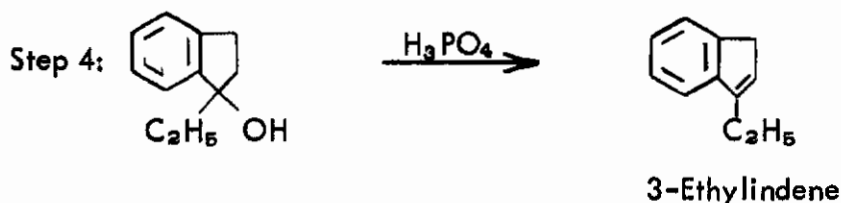
The solution of Grignard reagent (C_2H_5MgBr) was cooled to room temperature, and the liquid was decanted into a 125-ml Erlenmeyer flask, leaving the unreacted magnesium in the reaction flask.

The 1-indanone (3.6 g, 0.026 mole) was dissolved in 20 ml of ether. The flask containing the Grignard reagent was placed in an ice-water bath, and the indanone solution was added slowly, with vigorous stirring. A greenish-yellow precipitate formed.

Contrails



A dilute solution of sulfuric acid in ice water was poured into the mixture obtained from step 2, and it was stirred until all the solid had dissolved. The liquid formed two layers which were separated in a separatory funnel. The organic layer was washed several times with water, then it was dried with potassium carbonate. After drying, the ether was distilled off, leaving several milliliters of residue.



Ten milliliters of 85 percent phosphoric acid was added to the residue of step 3 (ethylindanol), and the mixture was heated to 50° C for about 15 minutes. A yellow precipitate formed. The organic phase was extracted with n-pentane. The pentane solution was washed with water, then filtered through glass wool into a 50-ml Erlenmeyer flask. The pentane was evaporated off, leaving about two milliliters of liquid residue. The product was stored under a nitrogen atmosphere.

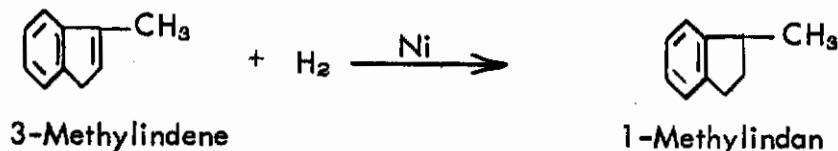
Analysis of product

The product was analyzed by gas chromatography, using a 2 meter by 1/4 inch column packed with Silicone 550 on a diatomaceous earth and a thermal conductivity detector. The column was operated at 150° C.

The chromatogram of the product showed three peaks, one for the solvents (pentane and ether), a small peak identified as 1-indanone, and a large peak with a retention time of 13 minutes. The material from this large peak was collected in a trap at -78° C as it came out of the chromatograph, and its infrared absorption spectrum was recorded. The IR spectrum matched the published spectrum of 3-ethylindene (5).

THE SYNTHESIS OF 1-METHYLINDAN AND 1-ETHYLINDAN

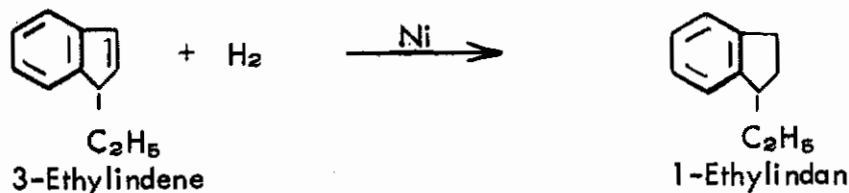
This organic preparation requires a corresponding indene as starting material. Some of these such as 1-methyl-1-indene are available from commercial sources. In other cases, the synthesis previously described for 3-ethylindene may be required as a preliminary step. Conversion of an alkylated indene to the corresponding indan is accomplished by a light hydrogenation treatment using a Raney nickel catalyst. The exact procedure for the preparation of 1-methylindan is described below.



One milliliter of the 3-methylindene was mixed with about 2 ml of n-heptane and about 1 ml of Raney nickel catalyst in a heptane slurry. This mixture was placed in a 5-ml stainless steel hydrogenation bomb, which was then mounted on a rocker apparatus. The bomb was flushed twice with hydrogen, then pressurized with hydrogen to 500 psi. The pressure was maintained at about 500 psi for approximately 2 hours -- until the pressure remained constant. The bomb was opened, and the mixture was centrifuged. The liquid was then decanted into a container, with the catalyst remaining in the centrifuge tube. The liquid was heated gently to evaporate most of the heptane.

The hydrogenated product was run on a vapor phase chromatograph, with a 2 meter by 1/4 inch aluminum column packed with Silicone 550 oil on diatomaceous earth. The column was operated at 150° C. Helium flowrate was 100 ml/min.

The retention time of the hydrogenated product was 5.2 minutes and the infrared spectrum matched the API spectrum of 1-methylindan. The exact procedure for preparation of 1-ethylindan is described below.



Contrails

One-half milliliter of 3-ethylindene was dissolved in about 2.5 ml of n-heptane, approximately 0.5 ml of Raney nickel catalyst was added, and the mixture was placed in a 5-ml stainless steel hydrogenation bomb. The bomb was mounted on a rocker apparatus and pressurized with hydrogen to about 1900 psi. The shaker was turned on, and it was left for 1½ hours. Then the mixture was poured out of the bomb, filtered through glass wool to remove the catalyst, and heated to evaporate the solvent.

The hydrogenated product was analyzed by vapor phase chromatography. The major component had a retention time of 9 minutes.

The major component of the hydrogenated product was trapped at -78° C and the infrared spectrum recorded. The spectrum was identical to the spectrum of the 3-ethylindan.

Security Classification

DOCUMENT CONTROL DATA - R&D		
<i>(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)</i>		
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13. ABSTRACT This report covers an investigation of the influence of fuel constituents during storage on thermal stability of Spec MIL-J-25656B type (JP-6) and other high temperature fuels. The major objectives of the program were satisfied by the successful development and evaluation of several methods utilizing radiotracers to determine the causes of thermal instability as well as predicting thermal stability quality of high temperature fuels during storage. Ninety-four test blends of 12 fuels and 9 radioactive fuel compounds were prepared and tested in the evaluation of a microscale thermal stability procedure developed for the purpose of determining the contribution of selected fuel components to thermally induced deposits. Eighty-eight of these test blends were retested after 26 weeks storage at 130° F to determine the effect of storage upon deposit-forming tendencies. Analysis of the data obtained in this study resulted in a further study of thermal decomposition of a fuel antioxidant additive. An investigation of several variables associated with an <u>in situ</u> hydrogenation method using tritium as the index of reactivity was completed as an effort to develop a sensitive and accurate analytical technique for very low concentrations of olefins in hydrocarbon fuels. In addition, initial steps were taken toward obtaining or synthesizing radioactive materials and reference standards essential to a continuation of this program along a similar line of approach.		

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Security Classification

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	ROLE	WT	ROLE	WT	ROLE	WT
Isotopes Fuel Storage Stability Jet Fuel Radioactive Labeling of Fuels Thermal Stability						

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