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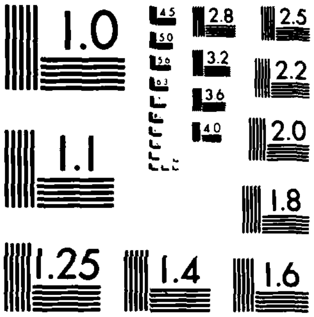

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Quantitative Determination of Compound Classes in Jet Turbine Fuels by High Performance Liquid Chromatography/Differential Refractive Index Detection

Part 2

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19 ABSTRACT (Continue on reverse if necessary and identify by block number)

Recent investigations ~~have~~ demonstrated the usefulness of high performance liquid chromatography/differential refractive index detection (HPLC/DRI) as a method for quantitating compound classes present in jet fuels. The method is fast, precise and accurate when applied to mixtures of known compounds typical of each compound class encountered in a fuel. It is also accurate when the refractive index of the fuels class and the detector calibration standard are matched. This study assesses the accuracy of HPLC/DRI when the calibration standards are pure compound blends whose refractive index for each compound class is matched closely to that of the fuels being analyzed. The accuracy of analyses by this method of calibration was checked by analyzing test fuels of known refractive indices. The test fuels were prepared by recombination of previously separated fuels after measuring the refractive index of each fraction.

The composition of fifteen fuels was also determined by the Fluorescent Indicator Absorption (FIA) method (ASTM-D-1319-77). In general the agreement between FIA and HPLC/DRI was quite good for many samples

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after conversion of the FIA aromatic content to weight percent. Four fuels, three of which have unusually high dicyclic aromatic content did not give good agreement between the two methods of analysis. The results demonstrate that it is possible to accurately calibrate the detector's response factors for each compound class by standards derived from pure compounds. To ensure that the accuracy of the results falls within the limits of the electronic integrator's precision, one needs to match the refractive index of the saturate fraction to within approximately 0.004 RI units. The ranges of refractive indices for all of the fractions indicate the need for alternate calibration methods that allow one to determine the RI of each class readily or do not require measurement of the analyte RI in order to maximize the accuracy of the method. For Navy JP-5, this method appears to be a suitable substitute for the FIA method.

CONTENTS

QUANTITATIVE DETERMINATION OF COMPOUND CLASSES IN JET TURBINE FUELS BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY/DIFFERENTIAL REFRACTIVE INDEX DETECTION. PART 2

INTRODUCTION

The effects of variations in composition on critical performance properties of JP-5 jet fuels are well documented [1,2]. Currently the results indicate that fuel hydrogen content may be the best single measure of overall fuel quality as related to combustion behavior. Since hydrogen content is directly related to a fuel's compound class composition, the ability to determine the compound class composition accurately should aid in establishing these performance/composition correlations. HPLC has been used to obtain quantitative data on jet fuels routinely in this laboratory [3-5], but until recently [6] it was coupled with some other technique such as gravimetric methods [3] or gas chromatography [5]. Other investigators [7-9] have applied HPLC/DRI to hydrocarbon class analysis. These studies have tried to obtain instrumentally the same characterization as one obtains with the ASTM FIA method [10] which separates the fuel into saturates, olefins and total aromatics. For relevance to jet fuel combustion, we have chosen to separate the fuel [6] into saturates-olefins, monocyclic aromatics, and dicyclic aromatics. Our reasoning for doing this is that response factors for monocyclic and dicyclic aromatics are significantly different from each other but relatively constant within each class [6]. Without this chromatographic separation, it is very difficult to calibrate the detector for the aromatic fraction since the response factor would change for different relative amounts of the two aromatic classes in the fuel. Therefore, one would essentially need a calibration standard for analyzing each fuel. In addition, the fuels of interest generally have very low olefin content and this can be determined by FIA if it is needed.

Initial investigations at NRL [6] demonstrated that HPLC/DRI can give precise and accurate quantitation of hydrocarbon mixtures over large concentration ranges when the chemical composition of the mixture is the same as the calibration standard used to determine the response factors for the detector. This calibration method is suitable for mixtures of pure compounds and fuel derived standards. In these examples the RI of the standard is essentially closely matched to each compound class of analyte.

The objectives of this work were several fold. First, to obtain samples of the three hydrocarbon classes for numerous jet fuels from many sources by preparative HPLC and to measure the refractive index of each fraction to determine the range of RI values to be expected for each compound class. Second, to select fuels that have been accurately analyzed previously and to reanalyze them with detector response factors obtained from a calibration standard prepared from pure compound mixtures

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blended to match the RI for each hydrocarbon. Third, to examine the effect on quantitation of mismatching the RI of the saturates over the full range expected. Fourth, to analyze each of these fuel samples by the FIA method in order to evaluate the HPLC/DRI results in a direct comparison with FIA.

EXPERIMENTAL

The HPLC system used in this study is outlined below. Separation was effected with a Whatman M-9 10/25 micron Partisil PAC semi-preparative column (chemically bonded alkyl amino-alkyl cyano)(9 mm I.D. by 25 cm). The pentane mobile phase was maintained at a flow rate of 6.0 ml/min with a Milton Roy Constametric pump operating in the 400-600 psi range. The injector was a Waters U6K with a 3 ml fixed loop. During quantitation, the sample size was maintained at approximately 25 µl to insure adequate chromatographic resolution while maintaining a sufficient signal to noise ratio to operate the Waters Prep 500 refractive index detector at a relative response setting of 5. Quantitation was achieved by calibration of the detector response with two types of calibration standards. The first was prepared by recombining weighed amounts of each compound class of a representative fuel, 82-17 (Calibration Standard 82-17). The second type used mixtures of pure hydrocarbons: dodecane and decalin represented the saturates class, 1,2,4-trimethylbenzene and tetralin represented the monoaromatics, and 1-methyl and 2-methylnaphthalene represented the di-aromatics. The relative amounts of the saturates and monocyclic aromatics were varied to give mixtures having the same RI for the respective fractions of the fuel to be analyzed. The composition of the dicyclic mixture was maintained at a 1:9 ratio for all calibration standards. The accuracy of the calibration was checked by preparing several "test" fuels by recombining weighed quantities of each compound class of some of the fuels used in this study. Integration of the detector output was obtained by a Hewlett-Packard 3390A Integrator (in parallel to a conventional recorder). The integrator was typically operated with the following run parameters: attenuator = 128x; chart speed = 0.50 cm/min; integrator peak width = 0.20 sec; baseline threshold = 6; and area rejection = 50,000 counts. When chromatographic fine structure was observed in the aromatic fractions, the "summation of peak areas" mode was used to integrate the total hydrocarbon class rather than the individual components. To collect samples for the refractive index measurements 100 µl of fuel was injected and the fractions were collected by observing the response of the refractive index detector on a conventional recorder. Typically a total of 2 ml of fuel was separated into fractions. The pentane was removed by warming at low heat on a standard hot plate. The purity of the fractions was checked with the HPLC system by injecting a small sample of the purified components. The saturate fractions required no further purification but the aromatic fractions required a second separation to remove the last traces of saturates which tailed into these fractions when the 100 microliter sample was injected. The refractive index of each collected class was measured on an Abbe refractometer. Although the refractometer was not thermostated, all of the results reported were obtained on the same day and a thermometer on the bench beside the refractometer remained constant within about 1°C at 21-22°C throughout the measurements. The samples were placed on the refractometer prism and a reading was immediately obtained. To insure the absence of solvent the prism was opened and the sample exposed to air for 1 min. The prism was closed again and a second reading was taken. This was repeated at 3 and 5 min intervals or until a constant RI reading was obtained. Generally, a constant reading was achieved at the three minute observation. The results reported represent the constant value obtained. A few of the samples had to be rewarmed for 30 min to 1 hr to remove solvent before constant readings could be achieved within five minutes. Some of the fuels contained so little dicyclics that it was not possible to obtain a RI reading for that fraction or it was not possible to follow the above procedure to obtain a constant value.

Each of the fuels was also analyzed by the current specification method for hydrocarbons in liquid petroleum products, ASTM D-1319-77, the Fluorescent Indicator Absorption (FIA) method. The procedure was followed carefully in order to compare our values to those of other workers where possible and to make it possible to compare FIA data to the HPLC/DRI data. The results of the FIA analysis for total aromatics were converted to weight percent by multiplying the FIA aromatic content by 1.08. This factor accounts for the density differences between hydrocarbon classes in JP-5.

RESULTS AND DISCUSSION

Fourteen fuel samples and one test fuel (82-17 test) were analyzed quantitatively for saturates, monocyclic aromatics and dicyclic aromatics by HPLC/DRI after calibrating the RI detector with Calibration Standard 82-17. This was done to evaluate the performance of the chromatographic system and to provide additional data to aid in evaluating long term analytical stability of the method. Also, operator effects on the results can be monitored since all the NRL data on the "83-" fuels were obtained with a different Prep 500 detector and a different operator. The results presented in Table I show very good agreement between the different analyses.

Table II lists the values of the refractive indices of the fuel fractions and each of the whole fuels. This data needs no explanation except to indicate that the refractive indices of the the saturate fractions of J-22 and 82-17 separated, purified and shown to be essentially pentane free by GC-MS gave identical refractive indices to the same fractions purified but not checked by GC-MS. Therefore it is reasonable to expect that the other values presented in this table also represent essentially pentane-free values.

In order to determine the significance of the small deviation in refractive index of each compound class in a given fuel from those in a fuel-derived calibration standard used to obtain the response factors, and to determine if calibration standards prepared from pure compounds could be substituted for fuel-derived standards, several new calibration standards were prepared. The first, Cal. Std.-A, was prepared to correspond to the measured refractive indices for the fuel J-22 since this fuel has the lowest RI value for the saturate fraction among the samples studied. Cal. Std.-A consists of a saturate fraction of dodecane and decalin blended to give a RI = 1.4315, a monocyclic fraction of 1,2,4-trimethylbenzene and tetralin with a RI = 1.5100 and the methylnaphthalene blend of RI = 1.6105. The detector was then recalibrated with Cal. Std.-A of known weight fractions of classes. The actual weight percents and the average measured composition determined for three trials of Cal. Std.-A are given in Table III. A test fuel was prepared by recombining fractions of J-22 previously separated with some of the methylnaphthalene mixture since not enough of the J-22 dicyclics were isolated. This mixture, J-22 Test, and fuel J-22 were then analyzed using the response factors obtained with Cal. Std.-A. Table III lists these results as well as the composition of J-22 as determined using Cal. Std.-82-17 described previously. This data clearly shows that it is possible to calibrate with pure compound mixtures. It is also noteworthy (J-22 data in Table III) that standardization with Cal. Std.-82-17 leads to low values for the saturates. This is because the saturate refractive index, which is higher for the 82-17 saturate fraction, leads to a lower response factor (amount/area). This type of experiment was repeated for fuels 82-17 and 81-14. Fuel 82-17 was chosen because good quantitation data were available for it and its saturate fraction had a typical RI. Sample 81-14 was selected because of the high RI value for its saturate fraction. Tables IV and V show the results obtained with these new calibration standards compared to the values determined with Cal. Std.-82-17. For 82-17 the results are the same regardless of the calibration standard, as expected, because both standards match the RI for the fuel. However for 81-14 the analysis gives higher values for the saturates with Cal. Std.-82-17. This is because the RI of the saturates in the standard are lower than those of 81-14 leading to a higher response factor (amount/area). The values determined with response factors derived with Cal. Std.-C should be more accurate for fuel 81-14 than those derived with Cal. Std.-B and Cal. Std.-82-17.

One other series of HPLC analyses were made. We compared the measured composition of the fuels 82-17 and 82-17 Test as determined with all of the different calibration standards.

Table VI shows that the results of the saturates analysis is high when Cal. Std.-A is used, low with Cal. Std.-C and the same with Cal. Std.-82-17 and Cal. Std.-B.

Thus we have demonstrated that accurate results can be achieved by HPLC/DRI when appropriate calibration standards are employed. In addition, the averages of the results of the analysis of the fuels in Table I are believed to be accurate to within ±1% for the saturate fractions. This is because Table I data were obtained using calibration factors obtained with an 82-17 calibration standard, a fuel with intermediate RI for the saturate fraction. The analysis of J-22 and 81-14 with Cal. Std.-A and Cal. Std.-C, respectively, are more accurate for those samples.

The final part of this study was to analyze each of these fuels by the FIA method to compare HPLC/DRI data to this specified method. Table VII lists the results of this work along with the standard deviation (S.D.) for each fuel. The S.D.'s were acceptable. These were all measured by the same operator using a single batch of silica gel, dye and alcohol to keep the conditions as constant as possible. These results are in general agreement with other FIA data that is available for five of the fuels. These agree with other work to within approximately one percent except for J-22 where our value is 2.4% lower than other data. Table VIII compares the FIA data converted to weight percent by multiplying the FIA volume percent by the factor 1.08. This factor accounts for differences in densities of the different classes and will vary depending slightly upon composition. The agreement is quite good except for four fuels: 81-13, 81-14, 81-17 and 83-43. Three of the four fuels showing poor agreement have unusually high dicyclic aromatic content which may have some adverse effect on the FIA analysis. These samples showing large discrepancies between FIA and HPLC/DRI are off-spec model fuels blended to afford a wide range of combustion properties.

CONCLUSIONS

The results of this study indicate that response factors for detector calibration generated from mixtures of pure compounds can provide accurate quantitation of hydrocarbon classes in JP-5 jet fuels. The method gives results within the limits of precision of the electronic integrator if the refractive index of the saturate fractions of the calibration standard and the fuel are within approximately ±0.004 RI units. Matching the RI's of the aromatic fractions is less critical since their DRI from pentane are two and three times as great as the saturates for the monocyclic and dicyclic compounds, respectively. Although one should try to match the RI of each class as closely as possible, proportionally larger ranges for the aromatic fractions should not effect larger absolute error in the accuracy. It should be pointed out that since the aromatic fractions represent less than 25% of JP-5, this would give larger relative errors for these fractions if the uncertainty in matching is greater than ±.004 RI units for these classes as well. Examination of Table II reveals that the refractive indices of the fuels studied remain remarkably constant for each compound class. If one prepares calibration standards with RI values equal to the average values of each class, then accurate quantitation near the limits of electronic integration precision should be achieved for this group of samples. There is no guarantee that future samples will fall within these narrow ranges and we are continuing this work to find alternate methods of determining response factors.

This study shows that HPLC/DRI is at least as accurate as the currently accepted FIA method of hydrocarbon analysis for JP-5 jet fuels. One sacrifices the olefin content to obtain a breakdown of the aromatics into the monocyclic and dicyclic fractions. Since current jet fuels contain relatively small amounts of olefins and the dicyclic content may be more important to know, this is not of great concern. A combination of FIA and HPLC/DRI would provide quantitative information on all four components. The HPLC/DRI technique appears to be superior to FIA in determining total aromatics in fuel blends containing unusually high concentrations (>3% w/w)of dicyclics.

TABLE 1

Comparison of Compound Class Quantitation Data from Various Sources*

Sample	Saturates	Monocyclic Aromatics	Dicyclic Aromatics	Method of Analysis
82-17-Test	78.1	17.5	4.5	HPLC-Cal.Std.-82-17(EUP)
82-17-Test	78.3	17.2	4.5	HPLC-Cal.Std.-82-17(NRL)
82-17-Test	77.9	17.5	4.5	Actual Weight %
J-22	75.0	24.2	0.8	HPLC-Cal.Std.-82-17(EUP)
J-22	75.1	23.9	0.9	HPLC-Cal.Std.-82-17(NRL)
J-22	76.5	23.3	0.3	LC-Gravimetric
81-14	76.5	16.5	7.0	HPLC-Cal.Std.-82-17(EUP)
81-14	76.2	16.2	7.6	HPLC-Cal.Std.-82-17(NRL)
82-17	76.3	18.4	5.3	HPLC-Cal.Std.-82-17(EUP)
82-17	76.4	18.3	5.3	HPLC-Cal.Std.-82-17(NRL)
81-19	75.5	23.8	0.7	HPLC-Cal.Std.-82-17(EUP)
81-19	74.8	24.5	0.8	HPLC-Cal.Std.-82-17(NRL)
81-17	68.5	22.6	8.9	HPLC-Cal.Std.-82-17(EUP)
81-17	68.7	22.8	8.5	HPLC-Cal.Std.-82-17(NRL)
81-13	72.1	24.1	3.8	HPLC-Cal.Std.-82-17(EUP)
81-13	71.8	24.3	3.9	HPLC-Cal.Std.-82-17(NRL)
81-15	77.0	19.6	3.3	HPLC-Cal.Std.-82-17(EUP)
81-15	77.2	20.1	2.8	HPLC-Cal.Std.-82-17(NRL
83-43	67.3	14.7	18.8	HPLC-Cal.Std.-82-17(EUP)
83-43	67.1	16.0	16.8	HPLC-Cal.Std.-82-17(NRL)
83-56	82.6	17.1	0.3	HPLC-Cal.Std.-82-17(EUP)
83-56	81.7	18.0	0.3	HPLC-Cal.Std.-82-17(NRL)
83-57	85.5	11.6	2.9	HPLC-Cal.Std.-82-17(EUP)
83-57	85.0	11.9	3.3	HPLC-Cal.Std.-82-17(NRL)
83-58	74.0	25.1	0.9	HPLC-Cal.Std.-82-17(EUP)
83-58	73.4	25.8	0.9	HPLC-Cal.Std.-82-17(NRL)
83-60	80.7	17.9	1.4	HPLC-Cal.Std.-82-17(EUP)
83-60	79.3	19.3	1.5	HPLC-Cal.Std.-82-17(NRL)
83-63	77.5	17.3	5.2	HPLC-Cal.Std.-82-17(EUP)
83-63	76.2	18.2	5.7	HPLC-Cal.Std.-82-17(NRL)
83-89**	82.7	16.8	0.4	HPLC-Cal.Std.-82-17(EUP)

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* All values are in weight percent.
EUP - Data obtained at Edinboro University of Pennsylvania
NRL - Data obtained at the Naval Research Laboratory
** Same as 83-56, later sampling from same drum

TABLE II

Refractive Indices of Compound Classes in Some JP-5 Jet Fuels and Jet Fuel Models

	FRACTION			
Fuel	Saturates	Monocyclic Aromatics	Dicyclic Aromatics	Whole Fuel
J-22	1.4318	1.5111	---*	1.4483
83-60	1.4363	1.5077	---*	1.4495
83-58	1.4376	1.5128	---*	1.4553
82-17	1.4376	1.5076	1.6080	1.4572
81-4	1.4378	1.5074	1.5950	1.4570
81-19	1.4382	1.5130	---*	1.4554
81-15	1.4385	1.5074	1.5902	1.4548
83-57	1.4387	1.5054	1.6065	1.4502
83-43	1.4402	1.5135	1.6006	1.4866
83-89	1.4405	1.5157	---*	1.4515
83-56	1.4405	1.5157	---*	1.4515
81-17	1.4405	1.5135	1.6008	1.4730
81-13	1.4413	1.5066	1.5902	1.4615
13-15	1.4414	ND	ND	ND
83-63	1.4415	1.5053	1.6000	1.4631
Pax River	1.4424	ND	ND	ND
81-14	1.4441	1.5100	1.5902	1.4642
Av.	1.4393	1.5102	1.5979	1.4586
S.D.	±0.0028	±0.0036	±0.0069	±0.0102
R.D.	±0.2%	±0.2%	±0.4%	±0.7%
Range	0.012	0.010	0.018	0.038

ND = Not Determined

* Insufficient material available for measurement.

All data at 22-23°C.

TABLE III
Comparison of Shale JP-5 Jet Fuel Class Quantitation Using Various Detector Calibration Standards

Sample	Saturates	Monocyclic Aromatics	Dicyclic Aromatics	Method of Analysis
Cal.Std.-A	71.7	22.2	6.1	Actual Weight %
Cal.Std.-A	75.0	20.0	5.0	Actual Volume %
Cal.Std.-A	71.6	22.2	6.2	HPLC-Cal.Std.-A
J-22-Test	71.5	22.1	6.4	Actual Weight %
J-22-Test	71.1	22.6	6.3	HPLC-Cal.Std.-A
J-22	76.3	23.0	0.7	HPLC-Cal.Std.-A
J-22	76.5	23.3	0.3	LC-Gravimetric
J-22	75.1	23.9	0.9	HPLC-Cal.Std. 82-17

TABLE IV
Comparison of Typical JP-5 Jet Fuel Class Quantitation Using Various Detector Calibration Standards

Sample	Saturates	Monocyclic Aromatics	Dicyclic Aromatics	Method of Analysis
Cal.Std.-B	74.9	20.4	4.8	Actual Weight %
Cal.Std.-B	74.9	20.4	4.8	HPLC-Cal.Std.-B
82-17	76.4	18.3	5.3	HPLC-Cal.Std. 82-17
82-17	76.2	18.9	4.9	HPLC-Cal.Std.-B

TABLE V
Comparison of Broadened Specification Test Blend Fuel Class Quantitation Using Various Detector Calibration Standards

Sample	Saturates	Monocyclic Aromatics	Dicyclic Aromatics	Method of Analysis
Cal.Std.-C	72.5	21.5	6.0	Actual Weight %
Cal.Std.-C	72.2	21.7	6.0	HPLC-Cal.Std.-C
81-14	75.4	17.9	6.8	HPLC-Cal.Std.-C
81-14	76.5	18.6	3.5	LC/GC
81-14	76.5	16.5	7.0	HPLC-Cal.Std.82-17

TABLE VI
Comparison of Results for Two Fuel Samples When Run Against a Series of Detector Calibration Standards Varying in RI

Sample	Saturates	Monocyclic Aromatics	Dicyclic Aromatics	Method of Analysis
82-17-Test	77.9	17.5	4.5	Actual Weight %
82-17-Test	78.3	17.2	4.5	Cal.Std. 82-17
82-17-Test	79.1	16.8	4.1	Cal.Std.-A
82-17-Test	77.7	17.9	4.4	Cal.Std.-B
82-17-Test	76.6	18.8	4.5	Cal.Std.-C
82-17	76.4	18.3	5.3	Cal.Std. 82-17
82-17	77.9	17.2	5.3	Cal.Std.-A
82-17	76.2	18.9	4.9	Cal.Std.-B
82-17	75.3	19.3	5.4	Cal.Std.-C

TABLE VII
Results of FIA Analysis of Jet Fuels

	Aromatics		Olefin		Saturates	
Sample #	%(Vol)	σ	%(Vol)	σ	%(Vol)	σ
83-57	13.5	0.33	1.2	0.18	85.3	0.30
83-89	16.3	0.37	1.3	0.30	82.4	0.44
83-56	16.7	0.24	1.2	0.19	82.1	0.32
83-60	18.0	0.42	1.3	0.26	80.7	0.34
83-63	21.3	0.22	1.5	0.25	77.2	0.19
J-22	21.6	0.30	1.4	0.22	76.8	0.70
82-17	22.1	0.39	1.0	0.08	76.9	0.40
81-15	22.7	0.53	1.6	0.21	75.7	0.41
83-58	22.9	0.17	1.3	0.26	75.8	0.15
81-19	22.9	0.19	1.1	0.22	76.0	0.05
81-14	26.2	0.41	1.6	0.33	72.2	0.39
81-13	31.4	0.57	1.8	0.54	66.8	0.91
81-17	32.8	0.30	1.1	0.13	66.1	0.15
83-43	40.7	0.41	0.8	0.17	58.6	0.43

TABLE VIII
Comparison of Results From FIA and HPLC/DRI Quantitation of Total Aromatics

Fuel	FIA		HPLC/DRI
	Vol. %	Wt. %*	Wt. %
J-22	21.6	23.3	23.7
81-13	31.4	33.9	28.0
81-14	26.2	28.3	23.7
81-15	22.7	24.5	22.9
81-17	32.8	35.4	31.4
81-19	22.9	24.7	24.9
82-17	22.1	23.9	23.7
83-43	40.7	43.9	32.8
83-56	16.7	18.0	17.9
83-57	13.5	14.6	14.7
83-58	22.9	24.7	26.3
83-60	18.0	19.4	20.0
83-63	21.3	23.0	23.1
83-69	16.3	17.6	17.3

* 1.08 Multiplication Factor

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