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Research on Detection Methods for Unconventional Pollutants in Ship Methanol Engine Exhaust Gas

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Hanqiao Zhang, Shanghai Marine Diesel Engine Research Institute

Hui Zhang, Shanghai Marine Diesel Engine Research Institute / National Key Laboratory of Marine Engine Science and Technology
Xianan Gu, Shanghai Marine Diesel Engine Research Institute

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ABSTRACT

Compared with ship diesel engines, ship methanol engines can significantly reduce the emissions of carbon smoke, nitrogen oxides, and sulfides in ship engine exhaust. However, there are many methanol and formaldehyde in the exhaust that are seriously harmful to the environment and human health, which need to be paid attention.The detection methods for methanol and formaldehyde can be divided into two categories based on the different detection principles: spectroscopic and chromatographic methods. Spectral methods include Fourier transform infrared spectroscopy, ultraviolet differential spectroscopy,laser infrared,etc. For chromatographic methods, there are gas chromatography and liquid chromatography, etc.The regulations of the US EPA/625/R-96/010b and the Chinese Ministry of Environmental Protection require the use of liquid chromatography to detect formaldehyde and gas chromatography-mass spectrometry to detect methanol for automotive methanol engines. This method is difficult to operate, and the sampling system for automotive engines is a full flow dilution system, making it difficult to directly apply in marine methanol engines.There is currently no unified and feasible standard regulation to guide the detection of methanol and formaldehyde in the exhaust of ship methanol engines.

This paper introduces the principles of various detection methods and conducts experimental verification of different detection methods based on the CS21DF-M marine methanol engine bench test, including the sampling and preprocessing methods of the sample gas, the stability and consistency of the test results, etc.The verification results show that spectroscopic method is simpler to operate compared to chromatographic method, and can achieve simultaneous online detection of methanol and formaldehyde in ship methanol engine exhaust. However, its measurement accuracy is slightly lower than chromatographic method, and the requirements for sample gas pretreatment method are more strict.This paper conducts research on sample gas pretreatment methods for the detection of methanol and formaldehyde in ship methanol engine exhaust based on spectroscopic methods, including the effects of different sampling tube materials, temperatures, lengths, and moisture contents on the test results. This provides data support for the use of spectroscopic methods for the detection of methanol and formaldehyde in ship methanol engine exhaust.

1 INTRODUCTION

Greenhouse gas emission reduction policies and regulations have become a major driver for the shipping industry, and methanol fuels are favored by the global transportation industry as one of the main alternative fuel choices for achieving the net-zero carbon emission target by 2050, with good economics, safety and accessibility.

Compared with marine diesel engines, methanol-fueled engines have significantly lower emissions of PM and NO_X and significantly higher emissions of HC, as well as unconventional pollutants such as methanol and formaldehyde in the exhaust.

Studies have shown that poor combustion of methanol engines will result in the presence of more methanol in the exhaust, while the oxidation of unburned HC in the cylinder and exhaust system will be converted to formaldehyde. Methanol and formaldehyde will produce serious harm to the environment and human body. At the same time, formaldehyde is also a greenhouse gas, and part of the atmospheric methanol in the photochemical reaction will also become formaldehyde. Therefore, from the point of view of the protection of the environment and human body, it is necessary to conduct relevant research on the emission of methanol and formaldehyde as the scale of methanol engine increases.

Aiming at the problem of unconventional pollutants in methanol-fueled engines, many enterprises and research institutions at home and abroad have carried out relevant research work. Xi'an Jiaotong University [1], Tianjin University [2], and Jilin University [3] in China have measured the unburned methanol and formaldehyde emissions from engines combusting methanol gasoline, mainly using gas chromatography. Chrysler, General Motors, Ford Motor Company, Mobil Oil Corporation [4], American Automotive Testing Laboratory, Beijing Institute of Technology [5], and others conducted batch testing of alternative fuel vehicles using full vehicle cycle conditions, and measured emissions of unconventional pollutants such as benzene, 1,3-butadiene, and formaldehyde by high-performance liquid chromatography (HPLC) and gas chromatography and mass spectrometry (GC-MS). Tsinghua University [6], AVL Austria, and the National Center for Technical Research of Finland [7] measured the transient emissions of unconventional pollutants from methanol gasoline vehicles under full vehicle cycle conditions using Fourier transform infrared (FTIR) spectrometry, with the main measurements of methanol, formaldehyde, and benzene. In general, the studies are relatively fragmented, with a long time span and lack of comprehensive and systematic testing and comparative analysis. Moreover, most of the current studies are focused on automotive methanol engines, and there are fewer studies on high-power marine methanol engines.

In this study, the principles of the existing detection methods and their feasibility in the application of high-power marine engines were analyzed, and the relevant verification was carried out in the CS21DF-M marine medium-speed methanol-fueled engine test bed, with a view to obtaining a method applicable to the detection of methanol formaldehyde in the exhaust gas of high-power marine methanol engines. The analysis of relevant influencing factors was also carried out to provide a basis for the subsequent exhaust gas testing and standardization of marine methanol-fueled engines.

2 INTRODUCTION TO METHANOL AND FORMALDEHYDE TESTING METHODS

2.1 Adsorption Column + HPLC/GC-MS

China's Ministry of Environmental Protection (MEP) released the "Measurement Methods for Non-Conventional Pollutant Emissions from Methanol-Fueled Vehicles [9]" on November 10, 2020, which specifies the measurement methods for formaldehyde and methanol in the exhaust of methanol-fueled light-duty automobiles, heavy-duty engines, and automobiles. The method utilizes filled column sampling tubes to collect methanol or formaldehyde from a full-flow dilution system, respectively, as shown in Figure 1, where the sampling tube for formaldehyde is filled with dinitrophenylhydrazine (DNPH) and the sampling tube for methanol is filled with silica gel adsorbent, which, in combination with a constant-flow sampling pump, enables the collection of methanol and formaldehyde from stationary dilution exhausts.

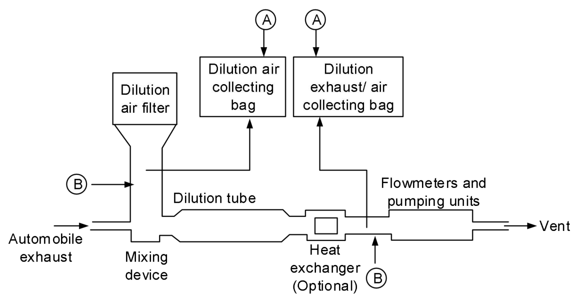


Figure1 Methanol/formaldehyde sampling schematics

After obtaining methanol and formaldehyde using a packed column sampling tube, the determination of formaldehyde concentration and methanol concentration can be realized by high performance liquid chromatography and gas chromatography-mass spectrometry, respectively.

The test method has been widely applied to the exhaust gas testing of vehicle engines, but there are the following problems in the real engine application of marine methanol-fueled engines:

(1) The power of marine methanol-fueled engines is much higher than that of automotive engines, so their exhaust flow is very large, and it is not possible to realize the full-flow dilution of exhaust, while the testing standards require that the quality of formaldehyde or methanol measured in the last sampling tube downstream should be less than 10.0% of the quality of the collection of all other upstream sampling tubes, so that only the direct extraction of exhaust and the tandem connection of multi-filled column sampling tubes can be used. After the verification of the real machine application, this method requires multiple sampling tubes to be connected in series, and is affected by the carbon fumes in the exhaust, the sampling tube resistance is larger, which is difficult to be applied in the real machine.

Figure 2. Adsorption column application diagram

(2) Using offline sampling, the measurement and analysis process of a single filled column sampling tube in the chromatograph takes about 30 minutes, which is time-consuming. During the development of marine methanol-fueled engines, it is difficult to provide timely measurement data of methanol and formaldehyde in the exhaust gas, and it is not able to support the parameter adjustment in the R&D process of the methanol machine in a timely manner.

Figure 3. HPLC/GC-MS detection procedure

2.2 Sampling bag + helium ion gas chromatography

Some scholars in China, such as Wei Yanju et al. [8] from Xi'an Jiaotong University, applied helium ion gas chromatography for the detection of unconventional pollutants in methanol formaldehyde, and its sampling process utilized a sample gas sampling bag for tail gas sampling, which was realized by the vacuum suction effect of the vacuum device for the sampling of tail gas from the methanol machine, and the specific sampling schematic diagrams are shown in Figure 4.

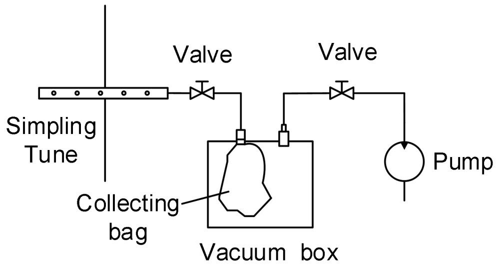


Figure 4. Vacuum sampling method schematic

After the gas in the sampling bag was pre-treated, the independent distinction of methanol and formaldehyde could be realized by using helium ion gas chromatograph and selecting suitable columns to achieve the simultaneous detection of methanol and formaldehyde, whose chromatograms are shown in Figure 5. At the same time, using helium ion detector, the rapid detection of methanol and formaldehyde in the exhaust gas of the methanol machine can be realized, and the analysis time of a single bag of sample gas is about 10 minutes, which makes the operation of this method simpler compared with the adsorption HPLC/GC-MS and greatly reduces the analysis cycle.

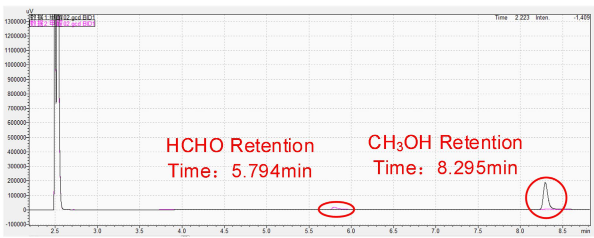
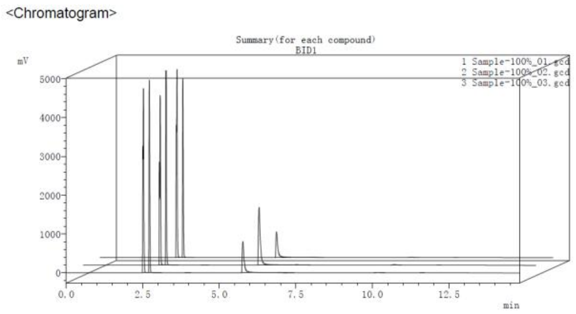


Figure 5. Methanol formaldehyde chromatogram

This test method has the following problems during the exhaust gas testing of marine methanol-fueled engines:

(1) Methanol machine exhaust compared to diesel exhaust, its water content is higher, the use of vacuum devices and sampling bags to collect

exhaust, high temperature gas into the sampling bag there is a process of condensation, methanol and formaldehyde and dissolved in water, so in the chromatograph analysis must be heated before the sample gas treatment, in the sample gas through the chromatograph is also required to keep the sample gas at a higher temperature, the sample gas pretreatment requirements are higher, and in the practical applications The repeatability is poor, and the results are shown in Figure 6 .



<< BID1 >>
ID#1 化合物名: CH3OH

Title	Sample Name	Sample ID	Retention Time	Aera	Height	Concentration
Sample-100%_01.gcd	Sample-100%		10.175	23658	3510	29.078
Sample-100%_02.gcd	Sample-100%		10.166	60969	9335	74.935
Sample-100%_03.gcd	Sample-100%		10.172	31144	4658	38.278
Average			10.171	38590	5834	47.430
%RSD			0.047	51.148	52.889	51.148
max			10.175	60969	9335	74.935
min			10.166	23658	3510	29.078
SD			0.005	19738	3086	24.260

Figure 6. Methanol formaldehyde detection repeatability verification

(2) The use of sampling bags to collect the exhaust gas from the methanol machine is limited by the volume of the sampling bags, which results in a smaller sample volume per sample, and the results of multiple sample bags collected under stable operating load conditions tend to vary widely. Table 1 shows the results of methanol and formaldehyde measured from multiple sampling bags collected under 100% load condition.

Table 1. 100% load condition multiple sample bag test results

Number	Formaldehyde (PPM)	Methanol (PPM)
1	54.6	87.3
2	46.5	92.9
3	64.6	108.4
4	52.0	137.3

2.3 Spectroscopy

Spectroscopic methods, which enable on-line detection of methanol and formaldehyde compared to chromatographic methods, mainly include Fourier Infrared Analysis, Differential Optical Absorption Spectroscopy and Laser Induced Fluorescence Spectroscopy. These methods often utilize the wavelengths or intensities of emission, absorption or scattering produced by molecules undergoing energy level jumps when material components interact with radiant energy for analysis.

The detection of methanol and formaldehyde in the exhaust gas of methanol engine using spectroscopic method can be realized by using the tracer sampling tube and pump to realize the continuous sampling of the exhaust sample gas, and the on-line detection of methanol and formaldehyde can be realized by the spectroscopic analysis equipment. This method is closer to the current stage of the detection of nitrogen oxides, carbon monoxide and other conventional emissions in the exhaust gas of ship engines, with real-time, efficient advantages, while most of the gas components can be detected, such as Fourier infrared analysis, theoretically more than 200 kinds of gas components can be detected.

At present, the mainstream engine exhaust testing equipment manufacturers, such as AVL, Horiba and CAI companies, have relevant spectroscopic principle of the testing equipment on sale, at this stage, this type of equipment is mainly used in the automotive gasoline engine or diesel exhaust N_2O or NH_3 detection, in the methanol-fueled engine exhaust in methanol and formaldehyde detection application of the study is still relatively small in the application process there may still be more factors affecting the results of methanol and formaldehyde measurement, such as the length of the heat pipe, temperature, exhaust gas moisture content, etc., need to carry out relevant research work to determine the method in the marine methanol fuel engine exhaust methanol and formaldehyde measurement results. In the application process, there may still be more factors affecting the measurement results of methanol and formaldehyde, such as the length and heat tracing temperature, the moisture content of the exhaust gas, etc. Relevant research work needs to be carried out in order to determine the applicability of this method in the detection of the exhaust gas of marine methanol-fueled engines.

3 FTIR UNCONVENTIONAL POLLUTANT FACTOR TESTING OF METHANOL FUELS

The FTIR equipment shown in Table 5 was utilized to verify the factors affecting different heat tracing pipe lengths, different heat tracing temperatures, and background spectrum, respectively.

3.1 Verification of Different Heat Tracing Pipe Lengths

The heat tracing pipe is mainly used to extract the sample gas, and carry out heat tracing and heat preservation for the sample gas during transmission and to avoid condensation of the components in the sample. The length of the heat tracing pipe is not suitable, which may cause the sample transmission process can not be in the appropriate temperature conditions, at the same time, for the strong adsorption of components, too long a pipe will increase the adsorption loss of the sample, resulting in the deviation of the measurement results. Therefore it is necessary to verify different heat tracing pipe lengths for methanol and formaldehyde.

The specific test process is as follows: After the FTIR equipment state is stabilized, the temperature of the detection unit and the heat tracing pipe reaches the factory-set temperature (190 ℃). The gas cylinder into the heat tracing pipe, and were passed into the 20, 40ppm formaldehyde, 400, 807ppm methanol, to ensure that the standard gas inlet pressure constant, respectively, access to 3m, 8m, 13m, 23m of the sampling tube for the test, and each group of conditions to record the stability of the data for 3min. The measurement results are shown in Table 2.

Table 2. Verification results for different heat tracing pipe lengths

gas	standard gas concentration	3m	8m	13m	23m
Formaldehyde (HCHO)	21	21.6	21.8	24.6	32.0
	40	41.9	41.8	46.3	49.5
Methanol (CH_3OH)	406	402.0	397.8	406.7	405.0
	807	799.1	795.5	806.9	806.6

According to the results, it can be seen that with the increase in the length of the heat tracing tube, the amount of formaldehyde measurement results deviation gradually grows, while the overall deviation of methanol is smaller, analyzed as the growth of the sampling length will strengthen the adsorption of formaldehyde and methanol. Formaldehyde due to the lower concentration of standard gas (high concentration of formaldehyde gas is easy to polymerize into paraformaldehyde, standard gas manufacturers prepare formaldehyde concentration is generally not more than 50 ppm), adsorption retention will be a higher impact on the low concentration of gas, sampling accompanied by the release of the residual gas will result in elevated measurement results.

Meanwhile, from 3m to 23m, the stabilization time of methanol is 10, 19, 27s and 62s respectively, the stabilization time increases greatly with the increase of length, in conclusion, it is not recommended to use too long heat tracing pipe for the test of unconventional pollutants in methanol fuel.

3.2 Verification of Different Heat Tracing Temperatures

Different heat tracing temperatures also have an impact on the accuracy of FTIR measurements. If the heat tracing temperature is too low, the gaseous components in the sample may condense. And methanol, formaldehyde components are soluble in water, will cause the measured concentration is lower than the actual value. The party accompanied by the heat pipe temperature is too high, for formaldehyde, such as containing aldehyde organic functional groups of compounds, too high a temperature will occur under the chemical changes, resulting in the measurement results can not be a true reflection of the actual emission situation. Therefore, it is necessary to verify the heat tracing temperature.

The specific test process is as follows: After the state of FTIR equipment is stabilized, the gas cylinder is connected to the sampling tube. As shown in Figure 7, adjust the temperature of the tracer tube as well as the pre-filter to 100 ℃ , 150℃, 190℃ (factory set temperature) and pass 21, 40ppm formaldehyde, 406, 807ppm methanol for testing, respectively, and record the stable data for 3min for each group of working conditions. The measurement results are shown in Table 3.

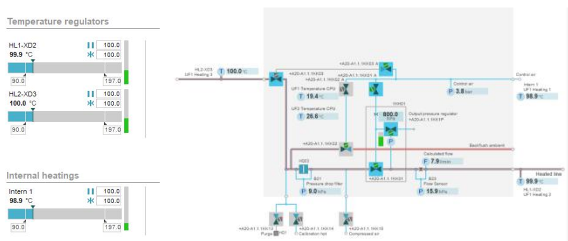


Figure 7. heat tracing temperature Setting

Table 3. Verification results for different heat tracing temperature

gas	standard gas concentration	100℃	150℃	190℃
Formaldehyde (HCHO)	21	21.7	22.2	21.6
	40	41.8	41.0	41.3
Methanol (CH_3OH)	406	407.7	407.8	406.7
	807	806.1	805.4	806.9

According to the results, it can be seen that in the measurement range, the heat tracing temperature has less influence on the test results of methanol and formaldehyde, and the default heat tracing temperature of 190 ℃ from the factory of the equipment has no influence on the testing of formaldehyde and methanol, compared with the requirement of 112℃±10℃ for the exhaust gas of methanol-fueled engines in the regulation of ISO8190 [10]. At the same time, it is not recommended to adopt the lower sampling temperature of ISO8190.

3.3 Verification of Background Spectrum

Before the FTIR test, the background spectrum must be deducted to correct the system spectral drift, to ensure that the zero point of the background spectrum is close to the factory calibration state. Due to the H_2O and CO_2 absorption peak range is large and many measurement gas region overlap, and equipment detection unit is easy to residual, easy to cause the background spectrum deduction of the spectral quality does not meet the requirements of the system, so the background spectrum determination needs to focus on the concentration of the two gases. Some equipment manufacturers will set the gas concentration limit, gas concentration exceeds the limit, can not perform the background deduction operation. In order to explore the background spectrum deduction, the background spectrum quality for methanol, formaldehyde measurement results of the degree of influence, this paper carried out a special verification.

The specific test process is as follows: When the FTIR equipment is just activated, the background spectrum will be in a poor state (H_2O > 40%, CO_2 > 40%, the percentage result is the deviation of the spectrum from the factory settings) due to the lack of dryness of the internal detection channel. At this time to modify the background spectrum limits, background spectrum calibration, using the standard port were introduced 21, 40ppm formaldehyde, 406, 807ppm methanol, test, each group of conditions recorded 2min stable data. After that, after a long period of blowing, the background spectrum was re-calibrated, at this time, the background spectrum meets the factory requirements (H_2O ≤ 20%, CO_2 ≤ 20%), and the above test process was repeated.

Table 4. Results of quality verification of different background spectrum

gas (i.e. gaseous substance)	standard gas concentration	Good background spectrum	Poor background spectrum
Formaldehyde (HCHO)	21	21.3	19.6
	40	41.8	41.0
Methanol (CH_3OH)	406	407.6	406.3
	807	807.0	825.4

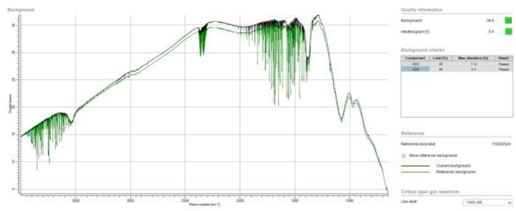


Figure 8. Good background spectrum (H2O:11.2%, CO2:2.4%)

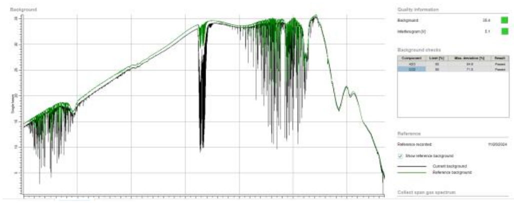


Figure 9. Poor background spectrum (H2O: 54.6%, CO2: 71.0%)

Before and after the two background spectrum calibration results are shown in Figures 8-9, the final results of the test are shown in Table 4, the background spectrum quality of the methanol, formaldehyde standard gas test results have less impact, analyze the reasons for this: although the actual environment is inconsistent with the factory calibration environment, resulting in spectral deviation. However, the standard gas has a single component and very low moisture content, and the calculation correction algorithm that comes with the equipment can eliminate the interference of methanol, absorption peaks and moisture background bias, while the absorption peaks of carbon dioxide are not close to each other with methanol and formaldehyde, so the accuracy has little impact.

However, considering that the water content in the exhaust gas of the actual machine is much larger than that of the standard gas, the combination of the background profile will cause a synergistic amplification of the error, superimposed on the formic acid and other components closer to the absorption peak of formaldehyde, which will have a greater impact on the accuracy of the measurements, so in practice it is best to follow the manufacturer's background profile limits, and then carry out the emission test.

4 ENGINE MEASUREMENT DATA

4.1 Engine Parameters

This study was carried out on a CS21DF-M turbocharged intercooled methanol engine, the model is currently in the research and development stage, and the main technical parameters of the engine are shown in Table 5.

Table 5. The main technical parameters of the test methanol engine.

Main technical parameters	unit	element
Diesel engine type		Inline six, turbocharged intercooled
Cylinder bore×stroke	mm x mm	210 x 320
Rated torque	N-m	11460
rated speed	r/min	1000
rating	kW	1200
fuel system		Methanol in-cylinder high pressure direct injection+ Diesel Pilot Ignition

4.2 Test Equipment

Using the I60FT Fourier analyzer from AVL, the device measurement accuracy is shown in Table 6.

Table 6. I60FT measurement accuracy

Non-conventional pollutant composition	range	Measurement accuracy
Methanol (CH3OH)	0-2000ppm	⩽ ±1%FS or 2% Reading, which ever less
Formaldehyde (HCHO)	0-1000ppm	⩽ ±1%FS or 2% Reading, which ever less

4.3 Test Results

4.3.1 Direct Data Collection

The test condition was the E3 propulsion cycle, each condition was stabilized for 10 min, and the last 3 min data were recorded. At the same time, the AVL I60 five-component emission analyzer was used to record the total hydrocarbon data simultaneously, which was used to examine the relationship between the methanol and formaldehyde concentrations and the hydrocarbon concentration. The test results are shown in Figure 10. During the working condition stabilization period, the methanol-formaldehyde gas concentration measured using the Fourier device was stable, with no abnormal fluctuation or obvious drift problems.

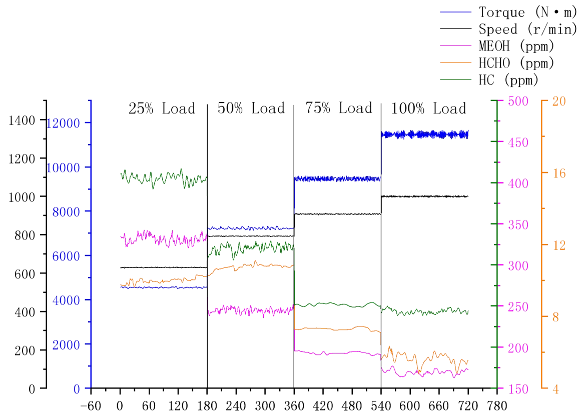


Figure 10. Non-conventional pollutants results

The average of the 3min results was also calculated and the results are shown in Table 7.

Table 7. E3 Non-Conventional pollutant emission Data

Load (%)	Substitution rate (%)	Methanol concentration (ppm)	formaldehyde concentration (ppm)	HC concentration (ppm)
25	79.0	336.1	9.3	406.1
50	82.9	244.0	10.4	321.2
75	84.6	191.5	7.3	252.5
100	90.1	170.0	5.6	246.4

4.3.2 Specific emission data

Calculation of non-conventional pollutant ratio emissions of methanol to formaldehyde with reference to ISO 8178.4:2020 regulation [10].

The carbon balance method was utilized to calculate the exhaust flow rate and brought into equation (1) for calculation:

$$q_{mgas} = u_{gas} \cdot c_{gas} \cdot q_{mew} \qquad (1)$$

where is the ratio of the density of each component of the exhaust to the density of the exhaust, 10^3, where under standard conditions (λ = 2, wet air, 273K, 101.3kPa), the density of methanol is 1.430kg/h, the density of formaldehyde is 1.340kg/h, the density of the exhaust of methanol fuel is 1.2610kg/m^3, and the density of the exhaust of diesel fuel is 1.2943kg/m^3, and the relevant mass flow rate is calculated according to the methanol as well as diesel fuel ratios, the relevant mass flow rate calculations were performed.

c_{gas} is the volume concentration of non-conventional pollutants, ppm, wet basis;

q_{mew} is the exhaust mass flow rate of the exhaust wet basis, kg/h, wet basis.

Carry out specific emission weighted calculation by using equation (2).

$$q_{mgas} = \frac{\sum_{i=1}^{n} q_{mgas,i} \cdot W_{F,i}}{\sum_{i=1}^{n} P_i \cdot W_{F,i}} \quad (2)$$

where $q_{mgas,i}$ is the mass flow rate of the unconventional gas in g/kW-h for each operating conditions;

WF_i is the weighting factor for each working condition.

The specific emissions of formaldehyde from this methanol machine were calculated to be 0.13 g/kW-h and the specific emissions of methanol were calculated to be 3.71 g/kW-h.

5 CONCLUSIONS

(1) For marine methanol engine, the use of adsorption column offline sampling there are adsorption column easy to clog, sampling difficulties, and vacuum sampling bag reheat analysis there are poor repeatability, and the use of on-line FTIR equipment is simple to operate, fast measurement speed, less sampling problems, and is more suitable for non-conventional pollutants in marine methanol engine detection

(2) Within the range of verification tests, the length of the heat tracing pipe has some influence on the detection results of unconventional gases, while the temperature and the quality of the background spectrum have little influence on the accuracy of the measurement results.

(3) The use of FTIR enables the testing of unconventional pollutants in marine methanol engines. Under the stabilized working condition of the real machine, the concentration of methanol formaldehyde gas measured by the equipment was stable, without abnormal fluctuation or obvious drift and other problems..

(4) For CS21DF-M high pressure methanol engine, the specific emission of formaldehyde is 0.13g/kW • h, and the specific emission of methanol is 3.71g/kW • h. Methanol and formaldehyde are the main components of HC pollutant in high-pressure methanol engine, accounting for about 70-80%.

6 DEFINITIONS, ACRONYMS, ABBREVIATIONS

PM: Particulate Matter

NO_X: Nitrogen Oxides

HC: Hydrocarbon Compounds

DNPH: Dinitrophenylhydrazine

GC: Gas Chromatograph

HPLC: High-performance Liquid Chromatograph

GC-MS: Gas Chromatography-Mass Spectrometry

FTIR：Fourier Transform Infrared Spectroscopy

H_2O: Water

CO_2: Carbon Dioxide

CH_3OH: Methanol

HCHO：Formaldehyde

E3: Test cycle for marine main engines and auxiliary engines operating on propulsion characteristics

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