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**Automobiles — Analytical procedure
for measurement of carbon monoxide,
carbon dioxide, total hydrocarbons
and oxides of nitrogen in exhaust gas**

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Foreword

This translation has been made based on the original Japanese Industrial Standard revised by the Minister of International Trade and Industry through deliberations at Japanese Industrial Standards Committee in accordance with the Industrial Standardization Law. Consequently JIS D 1030:1995 is replaced with JIS D 1030:1998.

In this revision, the measurement of hydrocarbons has been modified from non-dispersive infrared gas analyzer to hydrogen flame ionization detector which is effective for total hydrocarbons, and measurement for nitrogen oxide has been supplemented.

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Automobiles – Analytical procedure for measurement of carbon monoxide, carbon dioxide, total hydrocarbons and oxides of nitrogen in exhaust gas

1 Scope This Japanese Industrial Standard specifies, by means of using a non-dispersive infrared analyzer, hydrogen-flame ionization detector-type analyzer, or chemiluminescence analyzer, the analytical procedures for measurement of carbon monoxide, carbon dioxide, total hydrocarbons and oxides of nitrogen in exhaust gas, which are exhausted from such as gasoline automobile, diesel automobile, liquefied-petroleum-gas (LPG) automobile, or the engine mounted on those automobiles, owing to either method as the direct measuring method by which sampling is directly carried at an exhaust pipe or the dilution measuring method which is carried after diluting exhaust gas with air.

2 Normative references The following standards contain provisions which, through reference in this Standard, constitute provisions of this Standard. The most recent editions of the standards indicated below shall be applied.

JIS D 0101 *Glossary of terms relating to kinds of automobiles*

JIS D 0108 *Glossary of terms relating to control of air pollutions from automobile emissions*

JIS K 0055 *General rules for calibration method of gas analyzer*

JIS K 0211 *Technical terms for analytical chemistry (general part)*

JIS K 2240 *Liquefied petroleum gases*

JIS K 2249 *Crude petroleum and petroleum products—Determination of density and petroleum measurement tables based on a reference temperature (15 °C)*

3 Definitions For the purpose of this Standard, the definitions given in JIS D 0101, JIS D 0108, JIS K 0211, and the following definitions apply.

- a) **direct measuring method** The method by which exhaust gas is directly and continuously sampled from an exhaust duct using direct sampling apparatus and then the concentration and exhausted volume of target component in exhaust gas are measured.
- b) **dilution measuring method** The method by which exhaust gas is sampled after being diluted with air using constant volume-type sampling apparatus and then the concentration and exhausted volume of target component in diluted exhaust gas is measured.
- c) **diluted exhaust gas** The exhaust gas diluted with cleaned air using constant volume-type sampling apparatus.
- d) **total hydrocarbons** Organic compounds which are not-yet-burned fuels or already burned products contained in exhaust gas, and which can make response with a hydrogen-flame ionization-type analyzer. THC is its abbreviation.
- e) **concentration** Concentration is the volumetric ratio of carbon monoxide (CO), carbon dioxide (CO₂), total hydrocarbons (THC), and nitrogen oxide (NO_x) which are being contained in exhaust gas or diluted exhaust gas. The two type of concentrations measured

in wet exhaust gas and in dried exhaust gas are respectively used according to the serviceability. Generally, for CO the unit of vol ppm or vol% is used, for NO_x vol ppm, for THC vol ppmC, and for CO₂ vol%.

- f) **sampling apparatus** Apparatus for sampling and introducing it into an analyzer. Direct sampling apparatus, constant volume-type sampling apparatus, or other apparatus may be used.
- g) **direct sampling apparatus** Apparatus for directly sampling of a part of exhaust gas from exhaust pipe. This apparatus is used for direct measuring method.
- h) **constant volume-type sampling apparatus** The apparatus for sampling a part of diluted exhaust gas into sampling bag under definite flow ratio to the total flow rate, after the exhaust gas has been diluted with clean air so as to make the total flow rate of diluted exhaust gas become nearly constant. This apparatus is used for dilution measuring method.

The diluted exhaust gas, whose concentration is averaged while operating, can be obtained into the sampling bag. Sometimes the diluted exhaust gas may be continuously measured to avoid adsorption caused by temperature lowering of diluted exhaust gas. Generally, a fixed displacement-pump method (PDP method) or critical-flow venturi method (CFV method) is serviceable. It can be abbreviated as CVS apparatus.

- i) **NDIR** Among the apparatuses which measure the concentration of gas or liquid having absorption band at infrared region owing to absorption intensity of infrared at absorption wavelength, the non-dispersive infrared analyzer, which can select absorption band by a gas filter or selective detector instead of a prism or diffraction grating as in absorptionmetry, is abbreviated as NDIR.
- j) **FID** The hydrogen-flame ionization detector-type analyzer, which can obtain the concentration of total hydrocarbon (THC) owing to measuring ionization current having generated when sample was injected into hydrogen flame, is abbreviated as FID.
- k) **HFID** The heating-type hydrogen-flame ionization detector-type analyzer, whose both sampling route to detector and detector have been heated in order to avoid the condensation of water on the sampling route and the condensation and adsorption of hydrocarbons on the route, is abbreviated as HFID.
- l) **CL** The chemiluminescence analyzer, which can measure the concentration of nitrogen oxides (NO_x) making use of chemiluminescence taking place when nitrogen dioxide (NO₂) is produced by chemical reaction between nitrogen monoxide (NO) in sample and ozone (O₃), is abbreviated as CL.
- m) **HCL** The heating-type chemiluminescence analyzer, whose both sampling route to NO_x convertor and a detector and sampling route to detector have been heated in order to avoid the dissolution of nitrogen dioxide (NO₂) in condensed water, is abbreviated as HCL.
- n) **hang up** The phenomenon in which hydrocarbons staying in exhaust gas make an equilibrium condition by their adsorption and desorption on the flowing route for sampling and on the flowing route in analyzer. It may cause response delay.
- o) **tailing** The phenomenon in which response delays more than ordinary, showing like tailing, by the adsorption and desorption of hydrocarbons, in exhaust gas, on the flowing route for sampling and on the flowing route in analyzer.

- p) **NO_x convertor** Convertor by which nitrogen dioxide (NO₂) is changed into nitrogen monoxide (NO) for the purpose of measurement of nitrogen oxides (NO_x).
- q) **standard measurement range** When making measurement of concentration of exhaust gas, measurement range working as a standard for performance test of analyzer, which the range is selected from frequently used measurement ranges.
- r) **range switching accuracy** Reproducibility of measurements given within the measurement ranges.
- s) **bubbling** To confirm the degree of water interference in analyzer, zero gas and span gas shall be provided after bubbling in water.

4 Type of measuring methods For making the measurement of CO, CO₂, THC, and NO_x in exhaust gas or diluted exhaust gas, direct measuring method and dilution measuring method are serviceable.

According to the properties of targeted sample or to required test results, measuring method shall be chosen. Table 1 shows the purpose and type of measuring method, and Table 2 shows the type of an analyzer used at each method.

Table 1 Purpose and type of measuring method

Purpose	Applicable method
Measurement of concentration and calculation of exhausted volume of each component in exhaust gas	Direct measuring method
Measurement of concentration and calculation of exhausted volume of each component in diluted exhaust gas	Dilution measuring method

Table 2 Analyzer used at each method

Kind of automobile and engine	Component to be measured	Direct measuring method	Dilution measuring method
Gasoline • LPG	CO, CO ₂	NDIR	NDIR
	THC	FID	FID
	NO _x	CL or NDIR	CL
Diesel	CO, CO ₂	NDIR	NDIR
	THC	HFID	HFID ⁽¹⁾
	NO _x	HCL or NDIR	HCL, CL, or NDIR

Note ⁽¹⁾ Diluted exhaust gas shall be continuously measured.

5 Measuring apparatus

5.1 Construction Measuring apparatus is composed of a sampling apparatus, by which exhaust gas is introduced into an analyzer after necessary pretreatment, analyzer to measure the concentration of target component, and recording device for test results.

5.2 Sampling apparatus Sampling apparatus is as follows.

- a) **Direct sampling apparatus** Direct sampling apparatus is composed of, probe for sampling, introducing pipe for sample, filter, dehumidifier, pump for sampling, introducing inlet of calibration gas, and controlling system for sample flow rate. If necessary, heating device, pressure gauge, switching valve, or regulating valve of flow rate may be installed.
- 1) The construction of direct sampling apparatus and analyzer are exemplified in Fig. 1.1 and Fig. 1.2.
 - 2) The sampling route from sampling point to analyzer shall be constructed to get no stagnation of gas flow, and inside volume of the route be made as little as possible.
 - 3) The sampling route of FID may be heated so as to avoid the hang up. When THC in exhaust gas of diesel automobile is measured, the sampling route of HFID shall be heated up to from 160 °C to 200 °C.
 - 4) The sampling route of HCL and NDIR, to measure NO_x in exhaust gas of a diesel automobile or engine, shall be heated at least 60 °C from the probe of sampling to NO_x convertor in order to avoid the dissolution of NO_x in water.
 - 5) When influence by water in sample is not negligible, employ a dehumidifier. The dew point of the sample once having passed through the dehumidifier shall fall in the range from 0 °C to 7 °C.
- b) **Constant-volume type sampling apparatus (CVS apparatus)** The construction of constant-volume type sampling apparatus is as follows.
- 1) The construction of constant-volume type sampling apparatus is exemplified in Fig. 2.1 and Fig. 2.2.
 - 2) When multi-components are measured by dilution measuring method, the construction of flow route system and analyzer are exemplified in Fig. 3.
 - 3) The flow route from sampling point to analyzer shall be constructed to get no stagnation of gas flow, and inside volume of the route be made as little as possible.
 - 4) The sample for HFID shall be taken at the place where exhaust gas and air have been mixed sufficiently. When measurement is carried out using a CVS apparatus equipped with a heat exchanger, take sample at the point of upper stream side of the heat exchanger. Heat the sampling route of analyzer from 160 °C to 200 °C to eliminate the phenomena of hang up and tailing.

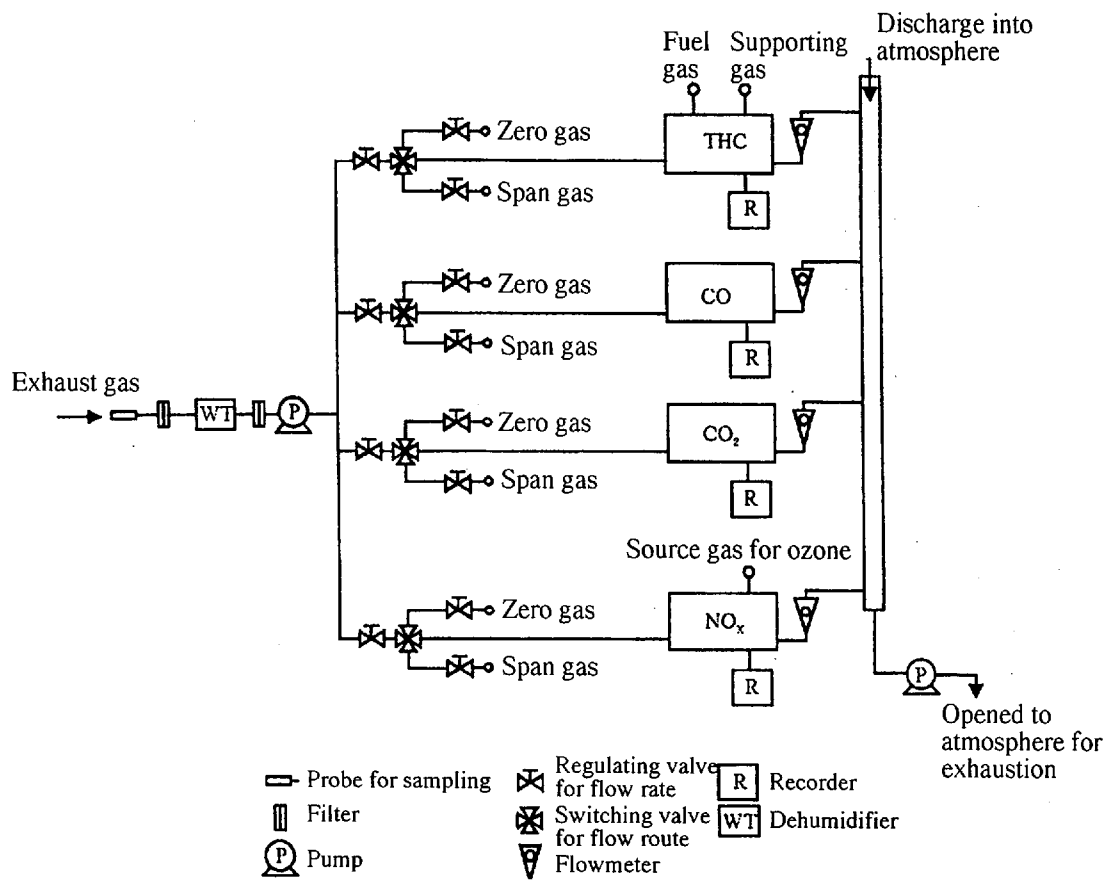


Fig. 1.1 Example of construction of direct sampling apparatus and analyzer (case of gasoline and LPG fuel)

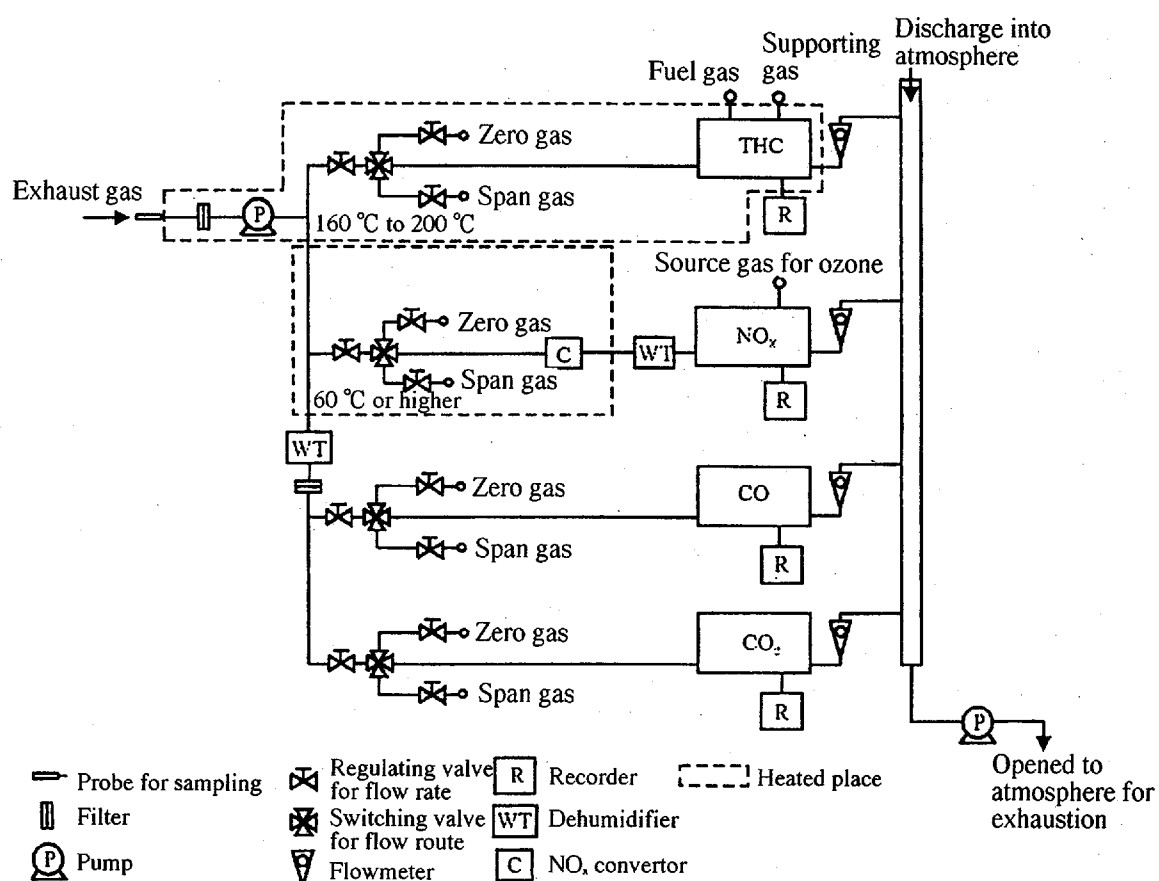


Fig. 1.2 Example of construction of direct sampling apparatus and analyzer (case of gas-oil fuel)

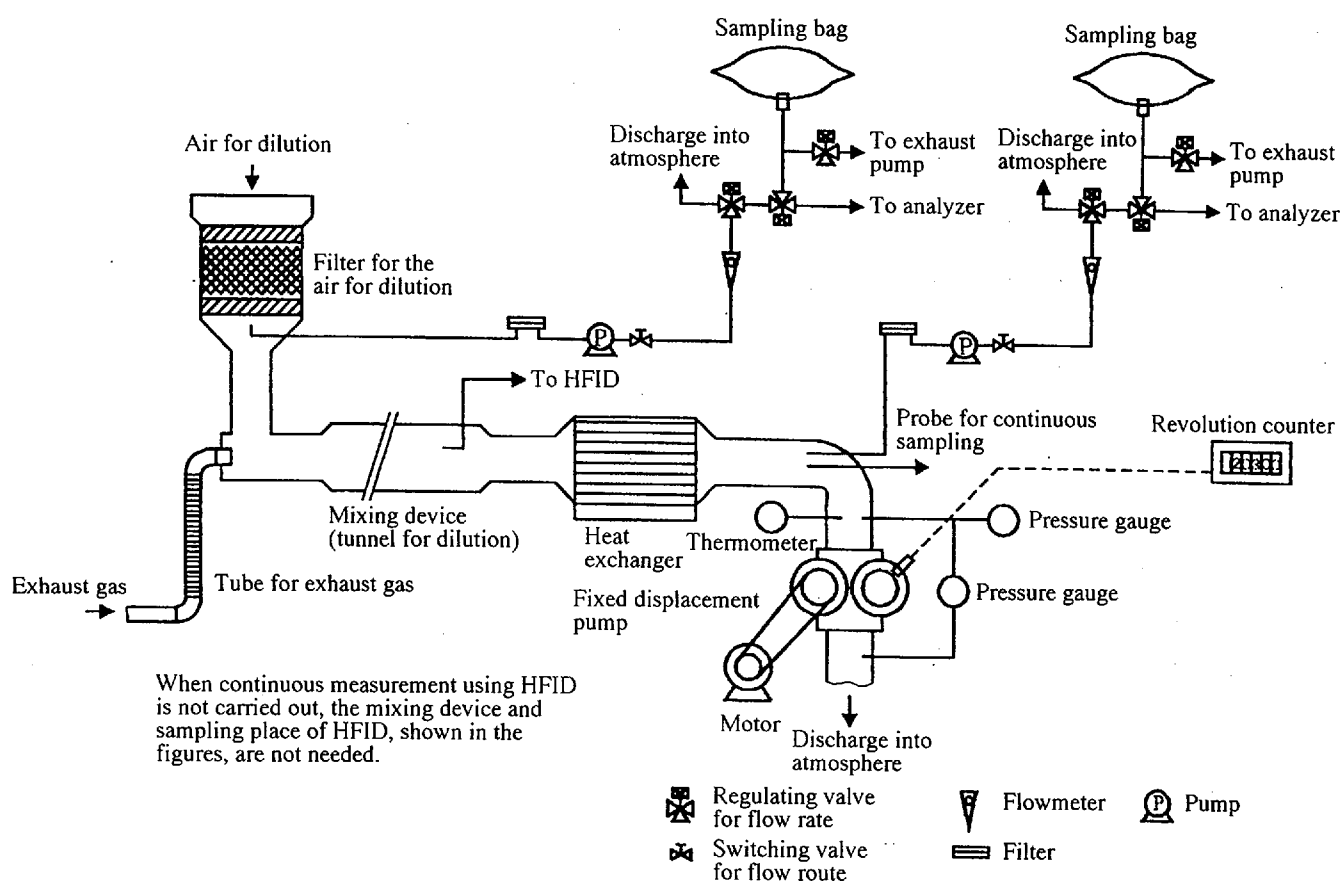


Fig. 2.1 Example of construction of constant-volume sampling apparatus (PDP method)

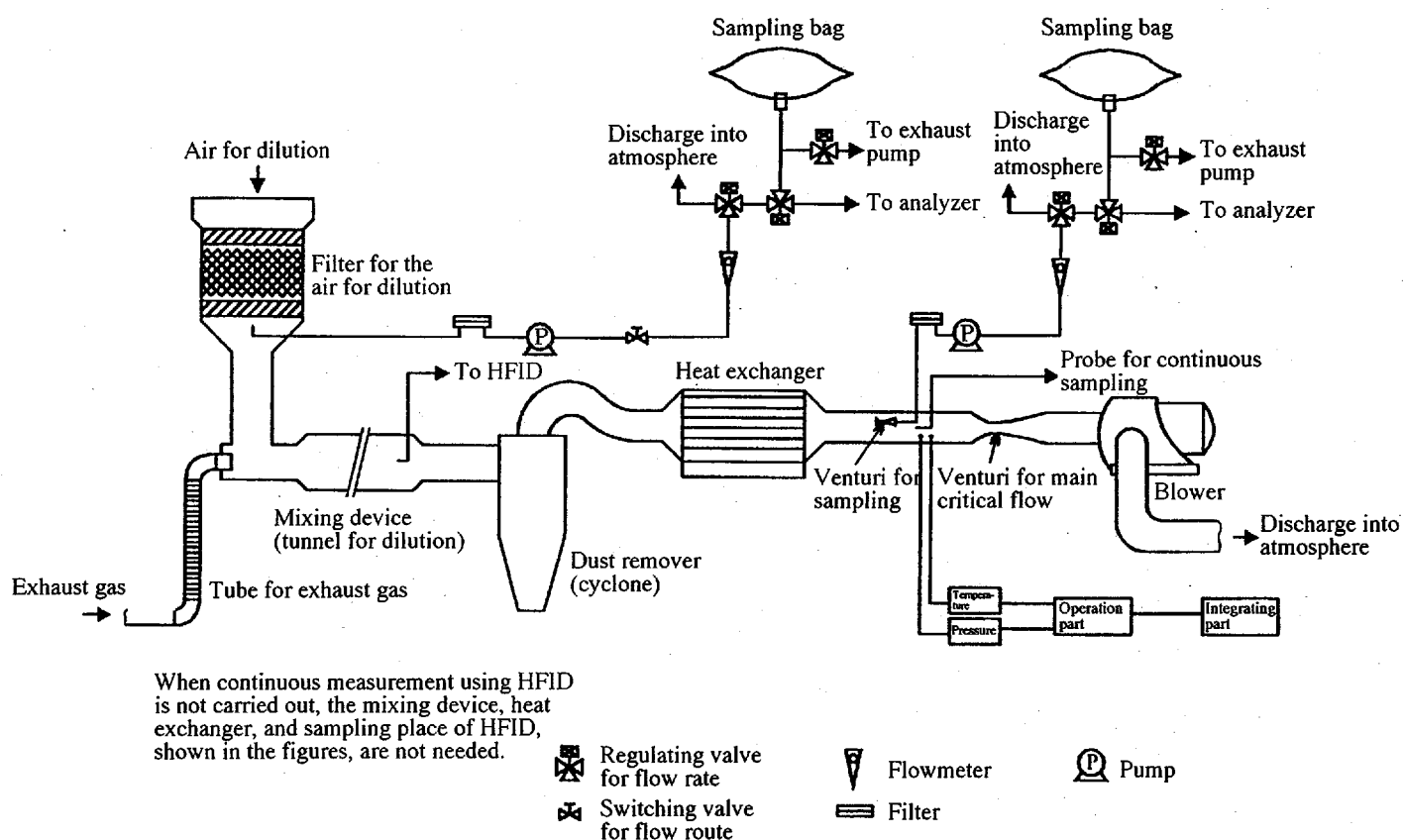


Fig. 2.2 Example of construction of constant-volume sampling apparatus (CFV method)

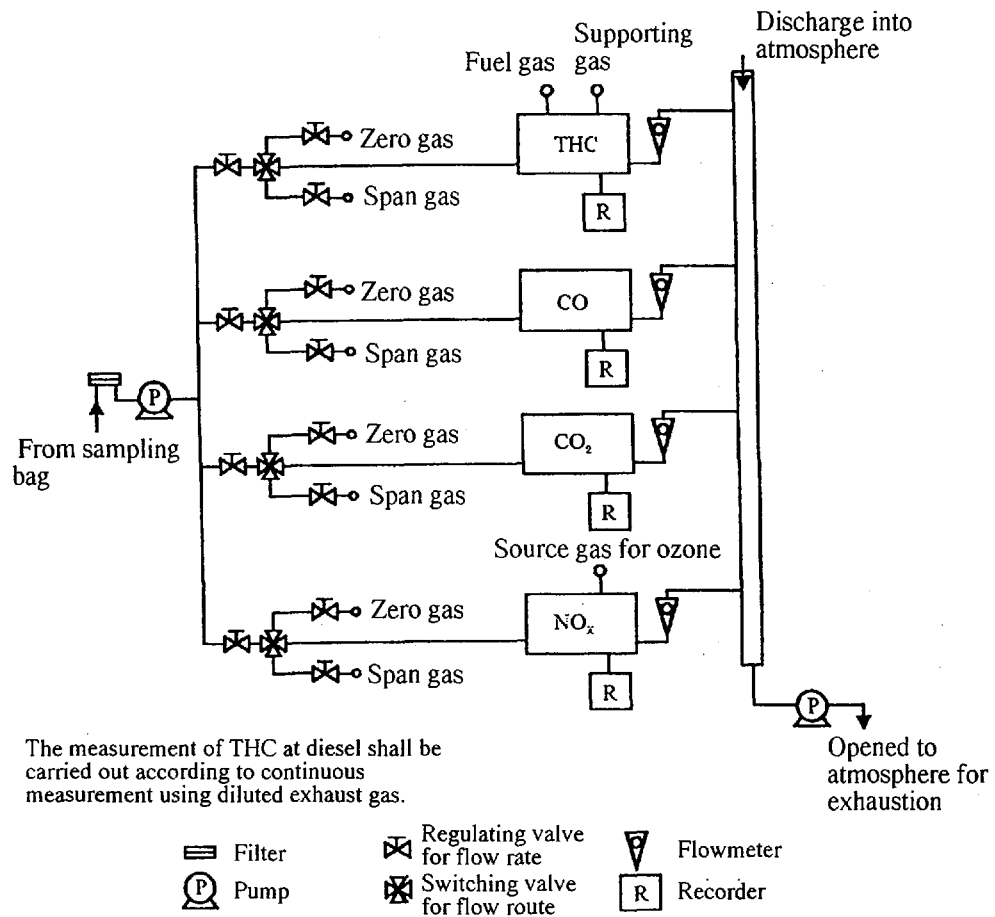


Fig. 3 Example of construction of flow route and analyzer when multi-components are measured using dilution measuring method

5.3 Analyzer Analyzer shall be as follows.

- a) **Limit of measurement range** The limit of measurement range of analyzer shall be as follows.
- 1) The limit of measurement range and standard measurement range, needed for analyzer, are shown in Table 3.
 - 2) The measurement range shall be measurable for sample at least 10 % or more of full scale. Such measurements, however, shall be eliminated as direct measurement containing transient running of automobile or engine, continuous measurement of diluted exhaust gas, and measurement of dilution air.
 - 3) If necessary, the analyzers whose measurement range is out of those shown in Table 3, may be used. In this case, it shall be considered that the operational performances of the analyzer are different from that shown in b).

Table 3 Limit of measurement range

Target component for measurement	Analyzer		Limit of measurement range	Standard measurement range
CO	NDIR	For dilution measurement	100 vol ppm to 3 000 vol ppm	300 vol ppm or its vicinity
		For direct measurement	0.5 vol % to 12 vol %	1 vol % or its vicinity
CO ₂	NDIR		1.0 vol % to 16 vol %	4 vol% or its vicinity (in case of dilution measurement)
				16 vol% or its vicinity (in case of direct measurement)
THC	FID or HFID		10 vol ppmC to 5 000 vol ppmC	100 vol ppmC or its vicinity
NO _x	CL or HCL		10 vol ppm to 5 000 vol ppm	100 vol ppm or its vicinity
	NDIR		200 vol ppm to 6 000 vol ppm	1 000 vol ppm or its vicinity

Note (²) The limit of measurement range shall not specify the maximum or minimum measurement range of each analyzer. The measurement range of each analyzer, which contains the maximum and the minimum value, can be arbitrarily established within the limit shown in Table 3.

b) **Operational performances of analyzer** The operational performances of analyzer shall be as follows.

1) **Standard value of operational performances** The operational performances of an analyzer, under the following test conditions, shall follow the values in Table 4, except the items respectively required by each analyzer. At other range than the limit of measurement range shown in Table 3, other value than that shown in Table 4 can be used as the standard value of operational performances.

- Measurement range : standard measurement range shown in Table 3
- Surrounding temperature : in the range from 5 °C to 35 °C and within ± 5 °C of fluctuation while testing
- Humidity : from 45 % to 85 % of relative humidity
- Atmospheric pressure : in the range from 95 kPa to 106 kPa and within ± 0.5 % of fluctuation while testing
- Voltage of power source : rated voltage $\pm (2 \text{ % or less})$
- Frequency of power source : rated frequency $\pm (0.2 \text{ % or less})$
- Route of gas flow shall be completely cleaned.

Table 4 Standard value of operational performances of analyzer

Items	Operational performances
Zero drift	± 1 % or less of full scale/8 hours
Span drift	± 1 % or less of full scale/8 hours
Reproducibility	± 1 % or less of full scale
Linearity	± 1 % or less of full scale (omit NDIR) ± 2 % or less of full scale (NDIR, but omit the analyzer having no linearizing function)
Response time for 90 % ⁽³⁾	2 seconds or less (omit NDIR) 5 seconds or less (NDIR)
Range switching accuracy ⁽⁴⁾	± 1 % or less of full scale
Duration for warming up	2 hours or less (omit NDIR) 8 hours or less (NDIR)
Surrounding condition	From 5 °C to 35 °C of temperature, from 45 % to 85 % of relative humidity

Notes ⁽³⁾ Duration from the instant when indicated value shows swinging after sample gas was introduced in the inlet of analyzer to the instant when the value gives 90 % of final indicated value.

⁽⁴⁾ Range switching accuracy contains the accuracy of zero point and the accuracy of decay value between two adjacent measurement ranges.

- 2) The test gas, for checking influences by interfering component when using an analyzer for CO gas, and its allowable limit are shown in Table 5.

Table 5 Test gas for checking influence by interfering component and its allowable limit

Interfering components		CO ₂ and water content
Test gas	Analyzer for dilution measurement	Nitrogen containing CO ₂ 3 vol% is treated with bubbling in water at room temperature (20 °C to 30 °C) ⁽⁵⁾ .
	Analyzer for direct measurement	Nitrogen containing CO ₂ 15 vol% is treated with bubbling in water at room temperature (20 °C to 30 °C) ⁽⁵⁾ .
Allowable limit	Less than 300 vol ppm	± 3 vol ppm or less
	300 vol ppm or more	± 1 % or less of full scale

Note ⁽⁵⁾ In case of the analyzer equipped with a dehumidifier on its sampling route, when making interference test, supply the test gas into the analyzer via the dehumidifier.

- 3) The test gas, for checking influences by interfering component when using an analyzer for CO₂ gas, and its allowable limit are shown in Table 6.

Table 6 Test gas for checking influence by interfering component and its allowable limit

Interfering components		Water content
Test gas		Nitrogen is treated with bubbling in water at room temperature (20 °C to 30 °C) (°).
Allowable limit	Less than 3 vol%	± 0.03 vol% or less
	3 vol% or more	± 1 % or less of full scale

- 4) The operational performance of the analyzer for THC shall be as follows.
- 4.1) Make the interference by oxygen as small as possible.
 - 4.2) The response due to hydrocarbon shall be near the sensitivity which has been prepared by making propane (C₃H₈) standard.
 - 4.3) The detector of HFID and the flowing route of sample to the detector shall be heated up from 160 °C to 200 °C .
- 5) The operational performance of the analyzer for NO_x shall be as follows.
- 5.1) The analyzer for NO_x shall be equipped with NO_x convertor.
 - 5.2) The keeping duration of sample in NO_x convertor shall be within 2 seconds and the conversion efficiency from NO₂ to NO shall be at least 95 %.
 - 5.3) The mechanism by which sample can bypass the NO_x convertor to reach directly the analyzer shall be installed.
 - 5.4) When the exhaust gas of diesel automobile is directly measured, either the sampling route to NO_x convertor or the detector and sampling route to the detector shall be heated higher than 60 °C .
 - 5.5) In case of using the analyzer for NO_x using CL or HCL, the interference caused by carbon dioxide shall be as small as possible.
 - 5.6) The test gas, for checking influences by interfering component when using the analyzer for NO_x equipped with NDIR, and its allowable limit are shown in Table 7.

Table 7 Test gas for checking influence by interfering component and its allowable limit

Interfering components		CO ₂	CO	Hydrocarbon	Water content
Test gas		CO ₂ 15 vol% Diluted with nitrogen	CO 5 vol% Diluted with nitrogen	C ₃ H ₈ 1 vol% Diluted with nitrogen	Nitrogen is treated with bubbling in water at room tem- perature (20 °C to 30 °C) (°).
Allow- able limit	Less than 500 vol ppm	± 5 vol ppm	± 5 vol ppm	± 5 vol ppm	± 5 vol ppm
	500 vol ppm or more	± 1 % of full scale	± 1 % of full scale	± 1 % of full scale	± 1 % of full scale

5.4 Recording device of data The recording device of data shall be as follows.

a) **Recorder**

- 1) Response time shall be 1 s or less when effective recording width is moved.
- 2) The effective recording width shall be 100 mm or more.
- 3) Feeding speed of paper shall include the range of 10 mm/min to 200 mm/min.

b) **Automatic recording device**

- 1) The measuring accuracy of automatic recording device shall be within 1 % of full scale.
- 2) The measuring frequency of the automatic recording device needed when transient running is carried out shall be 0.2 s or less.

6 Gas

6.1 General When the gas that has been filled in a compressed gas cylinder is used, the material of the cylinder directly contacting with the gas shall be anti-corrosive and little adsorptive. Adequate pressure-reducing valve and throttle valve are employed.

6.2 Gas for calibration Gas for calibration shall conform to the description in **JIS K 0055**, and be as follows.

- a) **Zero gas** Zero gas is high pure air or high pure nitrogen. The components coexisting in it and its allowable concentration are shown in Table 8.

Table 8 Coexisting components in zero gas and its allowable concentration

Coexisting components	Allowable concentration
CO	1 vol ppm or less
CO ₂	1 vol ppm or less
THC	1 vol ppmC or less
NO _x	0.1 vol ppm or less

The relation between target components and zero gas is as shown in Table 9.

Table 9 Target components • zero gas

Target components for measurement	Zero gas
CO	High pure nitrogen
CO ₂	High pure nitrogen
THC	High pure air High pure nitrogen (when using span gas diluted with nitrogen)
NO _x	High pure nitrogen

- b) **Span gas** The concentration of span gas corresponds to 70 % to 100 % of full scale of measurement range employed for calibration, and the component gas shown in Table 10, which has been diluted with high pure air or high pure nitrogen, shall be used.

Table 10 Component in span gas and dilution gas

Target component for measurement	Component gas	Dilution gas
CO	CO	High pure nitrogen
CO ₂	CO ₂	High pure nitrogen
THC	C ₃ H ₈	High pure air High pure nitrogen (when using span gas diluted with nitrogen)
NO _x	NO	High pure nitrogen

Note (6) Applicable only to direct measurement where gasoline is used for fuel.

- c) **Midway point gas** The steps of concentration of midway point gas for linearity check are about 20 %, 40 %, 60 %, and 80 % (or 15 %, 30 %, 60 %, 75 %, and 90 %) of full scale of measuring range that is to be checked.

6.3 Fuel gas Fuel gas for FID and HFID is the gas designated by the analyzer, and chosen among those as pure hydrogen and hydrogen that has been diluted with helium by 40 ± 2 vol %. In any case, the concentration of THC, contained as coexisting component, shall be 1 vol ppmC or less.

6.4 Supporting gas The supporting gas for FID and HFID is high pure air. The concentration of THC, contained as coexisting component, shall be 0.5 vol ppmC or less.

6.5 Source gas for ozone Ozone necessary for CL and HCL is prepared from the source of oxygen (O₂) or high pure air. The concentration of NO_x, contained as coexisting component, shall be 0.1 vol ppm or less, and the concentration of THC 10 vol ppmC or less.

7 Operations

7.1 General The preparation, checking, and measuring procedures of measuring apparatus shall follow from 7.2 to 7.5 and the method specified by manufacturer.

7.2 Preparation The preparation of measuring apparatus shall be as follows.

- a) **Condition for installation** Analyzers and compressed gas cylinders are preferably installed in such site as being satisfied with following conditions.
- 1) There is almost no vibration.
 - 2) There exists almost no corrosive gas or dusts.
 - 3) Humidity is not so high, and almost no temperature fluctuation.
 - 4) There is no direct sun shine.
 - 5) There is almost no fluctuation of power source voltage and its frequency.
 - 6) In order to make adsorption loss as little as possible, the length of sample introducing channel can be possibly short.
 - 7) There is no hindrance for maintenance.

- 8) The handling and setting of compressed gas cylinders shall conform to the High Pressure Gas Control Law.
 - 9) There is superior ventilation.
 - 10) The concentration of each target gas for measurement existing in air is sufficiently low and stable.
- b) **Preparation of measurement** The preparation of measurement shall be as follows. The preparation for calibration shall conform to this.
- 1) Check each part of measuring apparatus, and confirm there is no leakage of gas.
 - 2) Confirm that the flow rate and pressure of fuel gas and supporting gas for FID and HFID show specified values.
 - 3) Switch on power source sequentially as specified, and carry out warming up until each part gets stability.
 - 4) Confirm that, in case of CL and HCL, the flow rate and pressure of source gas for ozone, pressure in a reaction vessel, and the temperature of NO_x convertor show specified value.
 - 5) Confirm that the flow route for sampling of HFID and HCL has raised at specified temperature.

7.3 Zero and span adjustment Practicing frequency of zero and span adjustment shall be beforehand determined after investigating the maintenance period of its performance according to the type and serviceability of the analyzer. Generally, it is adjusted at beginning and ending time of measurement, and such interval as 8 h, 24 h, one week, or one month is chosen. Adjustment shall be as follows.

- a) Flow zero gas, adjust the indicated value on analyzer and recorder to zero at the lowest measuring range to be used, and confirm the zero point of each measuring range.
- b) Return it to the measuring range to be used, flow span gas, and adjust the indicated value of the analyzer and recorder to the concentration of span gas.
- c) Again, flow the zero gas, and confirm the zero point. If zero point falls out more than ± 1 % of full scale, try again the procedures in a) and b).

Remarks 1 In case of analyzer equipped with automatic calibration device, zero and span adjustment can be done with the automatic calibration.

2 When an automatic recorder is used, only analyzer can be used for adjustment.

7.4 Periodic check and calibration Practicing frequency of periodic check and calibration of measuring apparatus shall be beforehand determined after investigating the maintenance period of its performance according to the type and serviceability of the analyzer. Generally, the period shall be the term that does not exceed one year such as one month, 3 months, 6 months, or one year. Furthermore, it shall be carried out when the analyzer is installed or repaired. Periodic check and calibration shall be as follows.

- a) **Check of range-switching accuracy** Check the accuracy when range is switched. The example of the method is as follows.