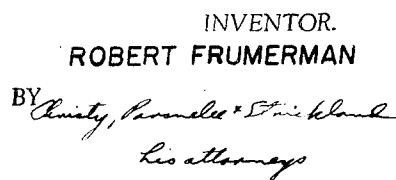


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IN A REFINING PROCESS
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METHOD AND APPARATUS FOR THE ANALYSIS OF OFF-GASES IN A REFINING PROCESS

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14 Claims

ABSTRACT OF THE DISCLOSURE

Method and apparatus for measuring the rate of flow of one constituent of a gas mixture and particularly where the one constituent is evolved from a mass of material undergoing chemical and/or physical change in a refining process or the like. A tracer gas having a predetermined known rate of flow is injected into and admixed with the off gas and the resulting mixture analysed to determine the fraction of tracer gas and the one constituent and a proportion established in order to compute the rate of flow of the one constituent.

While the invention is applicable to a number of processes, it will be described specifically in connection with the refining of steel. The term "refining" in this specific context refers to the removal of carbon from the molten steel. Increasing demands are made on the steel industry to produce steel containing carbon within narrower limits than was heretofore acceptable. Under older practices, the carbon content of the melt was determined principally by the development of a practice over a number of heats aided by time-consuming laboratory analysis of samples from the heat. This was satisfactory with a wider range of permissible carbon content in the final product and in older refining processes which permitted time for sampling and laboratory analysis. The advent of faster refining processes, such as the oxygen blast process, and the narrower permissible carbon limits requires new methods of carbon analysis if full advantage is to be taken from the newer refining methods.

The present invention provides a means for quickly and accurately determining the carbon content of the melt while refining is progressing, whereby an operator may accurately determine the desired end point. The specific embodiment illustrated is in connection with a vacuum degassing operation which in modern practices is generally the last refining step before casting the melt. The described embodiment of the invention contemplates first measuring the carbon content and weight of the melt at the start of degassing using known analytical methods, then continuously measuring the weight of carbon removed in the effluent gases and subtracting the latter quantity from the known initial weight of carbon to arrive at the carbon remaining in the melt. Substantially all the carbon removed from the melt is evolved as CO gas as is known in the art. This basic technique has already been suggested but until now there has been no accurate and economically feasible method or apparatus for analyzing the off-gases and relating the composition thereof to the composition of the melt nor is there any satisfactory way to directly and rapidly measure the carbon content of the melt. Some of the major problems encountered in the off-gas analysis include the difficulty of accurately measuring flow rates and the fact that the pressure in degassing system varies so that no simple analytical instrument can accept this variance without correction for pressure. Also, a gas sample at high vacuum which is compressed to atmospheric pressure before analysis, using a reasonable size of vacuum pump, be-

comes so small in volume that the time lag from pump discharge to analysis make the measurement non-representative.

According to the invention, off-gases from the degassing vessel are divided and a known small portion thereof passed through the sample system. Into the effluent gases there is injected a tracer gas at a known flow rate and the mixed off-gases and tracer gas are passed through two gas analyzers in a series at a controlled analyzer inlet pressure. One analyzer continuously delivers an output signal representative of the volume or mol fraction of CO in the sample and the other continuously gives an output signal representative of the volume or mol fraction of tracer gas in the sample. A signal indicative of the tracer gas input flow rate is continuously divided by the tracer gas analyzer output and the result continuously multiplied by the output signal from the CO analyzer to produce the instantaneous rate of CO evolution. The latter quantity is recorded and integrated to determine the total C evolved and this figure is subtracted from the initial C determination to find the C content of the melt at any given time. Stated differently, the ratio of mol fraction to volumetric flow rate for each of the CO and tracer gases is equal so that the unknown quantity, CO flow rate, can readily be determined from this proportion. Suitable pumps and pressure regulators are provided in the system to provide the necessary controlled flow conditions through the analyzers.

The invention utilizes the principle that the ratio of the partial pressures (mol fraction or other quantity derived from a volumetric analysis) of two constituents in a gas mixture is proportional to the ratio of the volumetric rates of flow of the two constituents into the mixture. Thus if the partial pressures of each constituent can be measured and the volume rate of flow of one constituent is known, the volume rate of flow of the other constituent can be calculated. This relationship is true regardless of the varying composition of the mixture. The partial pressure of a constituent is a function of the volumetric rate of flow of that constituent into the mixture. Other system variables, such as total pressure and the presence of other constituents in the mixture, are inherently taken into account in the established proportion since these variables affect all the constituents in the mixture in a proportionate manner. For the same reason, it is not necessary to pass all the tracer gas through the analyzers once it has been thoroughly mixed with the other constituents, and in fact in one embodiment of the invention much of the tracer gas in the described system for analyzing the effluent during degassing is not passed through the analyzers, but is discharged through the ejectors.

The invention therefore provides a new and useful method and apparatus for continuously determining the carbon content of a melt or for determining the quantity of a constituent remaining in a mass of material from which that constituent is evolved in gaseous form.

An object of the invention is to provide a new and useful method and apparatus for analyzing the gaseous effluent from a quantity of material undergoing a physical and/or chemical change.

Another object is to provide an improved method and apparatus for continuously determining the quantity of a constituent remaining in a mass of material from which that constituent is evolved in a gaseous form.

Another object is to provide an improved method and apparatus for continuously determining the amount of carbon remaining in a melt during a steel refining process.

Another object is to provide a method and a means for measuring the flow rate of one constituent of a mixture of gases flowing in a line, and particularly where the one constituent varies as a proportion of the mixture and the flow rate of the mixture also varies.

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These and other objects will be apparent to those skilled in the art and more fully understood by reference to the following description wherein:

The drawing schematically illustrates one embodiment of the novel apparatus for practicing the method of the invention.

Referring to the drawing, 10 is a container or ladle of molten carbon-containing steel in the process of being vacuum degassed. The degassing vessel 11 illustrated is a refractory lined vessel of the type having two depending tubes 12 and 13 with their ends submerged in the metal bath. A lifting gas such as argon or the like is introduced to the tube 12 through line 14 to aid in flowing metal upwardly into the vessel 11 where degassing takes place. The degassed metal flows downwardly through tube 13 back to the ladle. Vacuum conditions in the vessel 11 are created by ejectors 15 through line 16 communicating with the vessel 11. The off-gases in line 16 from vessel 11 comprise a mixture of gases including principally carbon monoxide and the lifting gas such as argon, smaller amounts of hydrogen and nitrogen, and negligible amounts of carbon dioxide and oxygen. A typical off-gas analysis (percent by volume) at the start and finish of degassing, using argon as a lifting gas, might be as follows:

	Start	Finish
Hydrogen.....	5	0.25
Carbon monoxide.....	Balance	Balance
Argon.....	10	50
Carbon dioxide.....	1	1
Nitrogen.....	5	0.25
Oxygen.....	1	1

Line 16, before mentioned, has a small length to diameter ratio on the order of 1:5 in order to carry the large volume of sub-atmospheric gases evolved from the metal in the degassing vessel. Paralleling line 16 is line 17 which has a large length to diameter ratio on the order of 10:1 and communicates with line 16 and the ejectors 15 through lines 18 and 19. Since streamline or laminar flow ordinarily exists in lines 16 and 17 at the low pressures therein, the flow in line 17 will remain a known fixed proportion of the flow in line 16. The mixture of gases carried by line 17 flows from junction 20 in two directions, through line 19 as before described and to line 21, then through vacuum pump 22, gas analyzers 23 and 24 and finally is discharged to atmosphere by vacuum pump 25, the flow being in the direction of the arrows in line 21. Various pressure control devices are placed in line 21 to regulate the pressure to the analyzers at about 5 mm. of Hg absolute.

The system shown is designed to supply a gas sample flow through the analyzers 23 and 24 of about one standard cubic foot per hour at a constant pressure of about 5 mm. of Hg absolute. At this pressure the flow is equal to about two actual cubic feet per minute. The analyzers illustrated and preferred in this system are infrared gas analyzers. The pressure level of 5 mm. is selected as a reasonable compromise since at this pressure the volume flow of sample gas is about two cubic feet per minute which is a reasonable flow for the analyzers to handle and pressure control is relatively simple compared to pressures in the higher vacuum range.

Since the pressure in the system, i.e., in line 17, varies between about one atmosphere down to 100 microns, two pressure control valves 26 and 27 in parallel are used to maintain the pressure at the suction side of pump 22 to a range of about 100 microns to 5 mm. Pressure control valve 26 in line 21 is paralleled by pressure control valve 27 in line 28, which communicates with line 21 on both sides of valve 26. Both valves 26 and 27 are controlled by pressure indicating controller 29, which monitors the pressure at the suction side of pump 22 and sends pneumatic signals through lines 30 and 31 to the control valves 26 and 27, respectively.

Intermediate the discharge side of pump 22 and the analyzer 23, by-pass line 32 is connected into line 21 and

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by-passes the analyzers 23 and 24, being connected at its opposite end to line 21 at the suction side of pump 25. Pressure control valve 33 in line 32 is controlled by pressure indicating controller 34 which senses the pressure at the discharge side of pump 22 and sends a pneumatic signal via line 35 to valve 33 to by-pass gases around the analyzers when the pressure at the discharge of pump 22 exceeds 5 mm. of Hg absolute, the desired pressure input to the analyzers. In line 21 downstream of line 32 and on the input side of analyzer 23 there is a differential pressure cell 36 which measures the flow into the analyzers and sends a pneumatic signal via line 37 to flow recording controller 38 which in turn sends a pneumatic signal via line 39 to flow control valve 40, which assists in maintaining the pressure in the analyzers by controlling the flow through them.

Because dirt must ordinarily be filtered from the effluent gases before passing through the analyzers a filter 22a is inserted in line 21 upstream of pump 22 to prevent contamination of the analyzers. A sintered stainless steel cartridge type filter has been found suitable, but other types may be employed. The filter should be one which does not introduce any appreciable additional time lag between sample point and analyzers.

The sample gas temperature at the analyzers should preferably be about 120° F. or lower. In the degassing installation illustrated this means that the gas temperature must be cooled from about 2900° F. down to 120° F. or lower. This heat is dissipated in the illustrated system by the pump 22. A Kinney model KDH-150 pump with a water jacket has been found suitable for this purpose. If required a separate cooler may be installed upstream of the analyzers. Ambient temperature and the temperature to the analyzers should not exceed 120° F. If higher ambient temperatures are encountered it may be desirable to physically locate the analyzers in an air-conditioned enclosure.

The tracer gas system is indicated generally as 41 and comprises a gas cylinder 42, which communicates with line 17 through line 43 and suitable pressure and flow control devices. The tracer gas is preferably introduced at an elbow in line 17 to promote mixing with the off-gases from the melt which are in laminar flow as before stated. The large length to diameter of line 17 also aids the mixing process, and the small diameter of the line reduces the amount of tracer gas required to achieve the desired result. While other gases could be used, nitrous oxide is preferred because it is readily detectable by infrared analyzers, is inexpensive and safe to use in this environment, and is substantially non-reactive with the other gases known to be present. The tracer gas pressure control system regulates the flow to about 4 to 5 pounds per hour and comprises a valve 44, pressure control valves 45 and 46 and flow control valve 47 all in line 43. Valve 46 is controlled by pressure recording controller 48 through pneumatic line 49. 50 is an electric resistance heater to control the temperature of the tracer gas in line 43. In the described system a constant temperature of about 165° F. has been found suitable. Intermediate valves 46 and 47 there is a differential pressure cell 51 which continuously sends a pneumatic signal indicative of the volume rate of flow of tracer gas to flow recording controller 52 through line 53. The output of flow controller 52 regulates valve 47 with a pneumatic signal via line 54 and this same signal, which is proportional to the square of tracer gas velocity through differential pressure cell 51, is sent to the square root extractor 55.

Referring back to the gas analyzers 23 and 24, analyzer 24 analyzes the sample gas flowing through it and continuously produces a pressure indicative electrical signal representative of the partial pressure or mol fraction of N_2O tracer gas in the sample. This electrical signal is sent through lead 56 to a transducer 57 which converts the signal to a pneumatic signal B which is sent via line 58 to divider 59 where it is combined with the pneumatic signal A from the square root extractor 55 via lead 60.

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At the same time CO analyzer 23 continuously produces an electrical signal representative of the partial pressure of CO in the sample stream and sends this signal via lead 61 to transducer 62 which converts it to a pneumatic signal C which is sent to a multiplier 63 via line 64. In the multiplier 63 the signal C is combined with the signal A/B from divider 59 via line 65. The multiplier continuously produces an output signal analogous to the computation

$$\frac{A}{B} \times C$$

where A is the rate of flow of N₂O tracer gas in standard cubic feet per minute, B is the mol fraction of N₂O tracer gas in the sample, and C is the mol fraction of CO gas in the sample, whereby the computation yields a signal D analogous to the instantaneous rate of flow of CO gas in standard cubic feet per minute. The resultant signal D equal to

$$\frac{A}{B} \times C$$

is sent via line 66 to an integrated flow recorder 67 and to an instantaneous flow recorder 68. Recorder 67 continuously integrates or totalizes the instantaneous signals D to provide an indication of the total amount of CO evolved from the start of the totalizing period. The totalized CO in the sample stream is readily convertible to totalized flow from the melt since the sample stream flow is a fixed known proportion of total flow from the melt. This figure is likewise readily convertible to total carbon flow from the melt, and when subtracted from the initial carbon content of the melt, yields the remaining weight of carbon in the melt. The percent by weight of carbon in the melt can readily be calculated, and this is the usual manner of expressing the carbon content.

The unique arrangement described regulates pressure in a manner to overcome the undesirable effects of pressure variations in the main stream and maintains the pressure to, and flow through, the analyzers substantially constant. Furthermore, even if the pressure at the inlet to the analyzers vary slightly from the control point, the mathematical utilization of the signals therefrom is such that the error due to pressure variations is minimized. The reason for this is that the outputs of the two analyzers are used as a ratio so that pressure errors tend to cancel each other out. If further pressure corrections should be desired the analyzer outputs can be passed through an analogue computer or the like to correct for deviations in pressure from the control point.

The conversion of the quantity of CO removed to the quantity of carbon removed can be quickly calculated by the operator using a graph or special slide rule or the like. Likewise, the weight or percentage by weight of carbon remaining in the melt may be obtained by integrating the instantaneous time rate of change of CO evolution using an analog computer or the like.

The gas analyzers preferably should be calibrated daily and for this purpose there is provided an analyzer output indicating meter 69 which selectively indicates the output of one or the other of the analyzers 23 and 24. Switch 70 is selectively positionable to connect the pressure signals from the analyzers to the indicating meter 69. Likewise the tracer gas bottle contents should be analyzed carefully and verified by the use of a standard gas bottle which has been carefully analyzed

The described embodiment of the invention thus comprises, by way of summary, a method and means for determining the carbon content of a melt of steel at any given time during the refining thereof, knowing the carbon content and weight of melt at the start of the refining process. The use of a tracer gas injected into the sample stream of off-gases at a known rate of flow of CO from

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the melt. The sample stream is analyzed for the mol fraction of CO and tracer gas and the signals developed thereby are combined with the flow rate of tracer gas to arrive at the flow rate of CO.

In the described embodiment laminar flow is assumed to exist in lines 16 and 17 and the tracer gas is introduced in line 17. In cases where turbulent flow is encountered, the tracer gas may be introduced into the degassing vessel itself and a sample stream taken off at some convenient point upstream of the ejector jets.

While the invention has been particularly described in connection with off gas analysis in a metal refining process, it is apparent that its usefulness may be extended to many other situations where it is desired to measure the flow rate of a constituent of a gas mixture and particularly where the flow rate of the mixture varies as well as the quantity of the constituent in the mixture. In the degassing process described, for example, pressure in the system and the gas flow may vary considerably during the process and the composition of the effluent gas mixture also varies considerably.

It will be apparent to those skilled in the art that various modifications of the method and apparatus described are possible within the scope and spirit of the invention.

I claim:

1. The method for determining the amount of carbon evolved from a mass of molten metal subjected to a refining process wherein carbon is continuously evolved from the metal substantially entirely in the form of carbon monoxide gas, comprising
 - (a) initially determining the carbon content of the metal,
 - (b) confining the evolved carbon monoxide gas to flow in a line,
 - (c) continuously injecting a tracer gas into the line at a known flow rate to effect mixing thereof with the carbon monoxide,
 - (d) continuously generating a signal analogous to the tracer gas flow rate,
 - (e) continuously analyzing the mixture in the line and generating signals analogous to the fraction of carbon monoxide and the fraction of tracer gas in the analyzed mixture,
 - (f) continuously combining the generated signals to produce output signals indicative of the flow rate of carbon monoxide, and
 - (g) totalizing the output signals to find the total carbon evolved.
2. The method for determining the amount of carbon evolved from a mass of molten metal in the form of carbon monoxide gas, wherein the carbon is evolved at a non-linear rate and in a mixture containing other gases, comprising
 - (a) confining the evolved mixture of gases to flow in a first line,
 - (b) dividing the flow to cause a known portion of the gases to flow in a second line having a substantially larger length to diameter ratio than the first line,
 - (c) continuously injecting a tracer gas into the second line at a known flow rate and effecting mixing thereof with the evolved gas mixture to form a sample mixture,
 - (d) continuously analyzing the sample mixture to determine the fractions of carbon monoxide and tracer gas in the sample mixture,
 - (e) continuously generating signals indicative of the tracer gas flow rate and the fractions of carbon monoxide and tracer gas in the sample mixture,
 - (f) continuously combining the generated signals in a manner to produce an output signal indicative of the flow rate of carbon monoxide, and
 - (g) totalizing the output signals to find the total amount of carbon evolved.
3. The method as defined in claim 2, wherein the tracer gas is nitrous oxide.

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4. The method as defined in claim 2, wherein the generated signals are combined to produce the output signal by continuously solving the equation

$$\frac{A}{B} \times C = D$$

where

- A is the rate of flow of tracer gas in volume per unit of time,
- B is the fraction of tracer gas in the sample mixture,
- C is the fraction of carbon monoxide gas in the sample mixture, and
- D is the rate of flow of carbon monoxide gas in volume per unit of time.

5. Apparatus for determining the rate of flow of a gas in a line, comprising

- (a) means for continuously injecting a tracer gas into the line at a known rate of flow,
- (b) gas analyzing means in the line capable of continuously generating signals indicative of the instantaneous fractions of each of the gas and tracer gas passing therethrough,
- (c) means for continuously generating signals indicative of the instantaneous flow rate of tracer gas, and
- (d) means for continuously combining the generated signals in a manner to produce an output signal indicative of the flow rate of the gas.

6. Apparatus as defined in claim 5, wherein the gas analyzing means comprises two analyzers in series, one for each of the gas and tracer gas.

7. Apparatus as defined in claim 6 wherein the two analyzers are infrared analyzers.

8. Apparatus as defined in claim 5, including pressure regulating means in the line, intermediate the point of injection of the tracer gas and the gas analyzing means, for maintaining a predetermined pressure at the input to the analyzing means.

9. Apparatus for determining the flow rate of a constituent of a gas mixture confined to flow in a line, comprising

- (a) a supply of tracer gas,
- (b) means for continuously introducing the tracer gas into the line to form a second mixture with the first mentioned mixture,
- (c) a pair of gas analyzers in the line downstream of the point of introduction of tracer gas, which analyzers are arranged in series to receive a flow therethrough of a regulatable sample stream portion of the total gas mixture flow, one analyzer being tuned to analyze the mixture for the fraction of tracer gas therein and the other being tuned to analyze the mixture for the fraction of the constituent therein, both analyzers being adapted to continuously generate signals indicative of the instantaneous fractions of their respective analyzed gases,
- (d) means of continuously generating signals indicative of the instantaneous flow rate of tracer gas,
- (e) means for regulating and controlling the amount of tracer gas introduced,
- (f) means in the line for regulating and controlling the flow through the analyzers, and
- (g) means for combining the generated signals to continuously produce output signals indicative of the instantaneous flow rate of the constituent.

10. Apparatus for determining the amount of carbon evolved from a bath of molten metal during a refining process wherein the carbon is continuously evolved in a gaseous state from a container of metal, comprising

- (a) a first line in which the evolved gas is confined to flow,
- (b) a second line smaller than and communicating with the first line and adapted to conduct a sample stream portion of the gas, which portion is a known proportion of the flow in the first line,
- (c) a container of tracer gas,

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(d) means for continuously introducing the tracer gas into the sample stream at a known flow rate,

(e) means for continuously generating signals indicative of the tracer gas instantaneous flow rate,

(f) a pair of gas analyzers arranged in series in the second line and through which the sample stream and tracer gas flow, one of the analyzers being adapted to continuously generate signals analogous to the instantaneous fraction of evolved carbon flowing therethrough and the other analyzer being adapted to continuously generate signals analogous to the instantaneous fraction of tracer gas flowing therethrough,

(g) means for continuously combining the generated signals to continuously generate output signals analogous to the instantaneous rate of carbon evolution, and

(h) means for continuously totalizing the output signals to compute the total amount of carbon evolved from the start of the totalizing period.

11. In a vacuum degassing installation having a container of carbon-containing molten metal to be degassed, a vacuum vessel through which the metal is flowed and while flowing therethrough carbon is evolved in the form of carbon monoxide gas along with other gases in a mixture, and an outlet duct in which the evolved gases are confined to flow, apparatus for measuring the amount of carbon evolved from the melt in the form of carbon monoxide gas, comprising,

(a) a pipe line communicating with the outlet duct and so arranged that a known portion of the evolved gas mixture flows therethrough,

(b) means for introducing a tracer gas into the evolved mixture to effect mixing thereof with the evolved mixture to form a sample stream mixture,

(c) means for regulating the introduction of tracer gas to a known flow rate, and

(d) means communicating with the pipe line for continuously analyzing the sample stream to continuously determine the respective instantaneous fractions of carbon monoxide and tracer gas flowing in the sample stream,

(e) means for continuously generating signals indicative of tracer gas flow rate and the fractions of tracer gas and carbon monoxide,

(f) means for continuously combining the generated signals to generate an output signal indicative of the instantaneous flow rate of carbon monoxide, and

(g) means for totalizing the output signals to indicate the amount of carbon monoxide evolved.

12. Apparatus as defined in claim 11 including means in the pipe line for filtering dirt from the evolved mixture before the mixture flows through the analyzing means.

13. Apparatus as defined in claim 11 including means for controlling the temperature of the gas mixture flowing into the analyzing means.

14. In combination with a container of carbon containing molten steel, a vacuum degassing vessel communicating with the metal, means for effecting flow of the metal into the vessel, an outlet duct communicating with the vessel for carrying away gases evolved from the metal, which gases contain carbon monoxide in a mixture with other gases from the metal, apparatus for measuring the amount of carbon monoxide evolved from the melt over a period of time, comprising

(a) a first pipe line communicating with the outlet duct and so arranged that a known portion of the evolved gases flow therethrough,

(b) a supply of tracer gas different from any of the other gases in the mixture which are present in substantial quantity and being substantially non-reactive with the other gases in the mixture,

(c) a second pipe line communicating with the first

- pipe line and the supply of tracer gas for introducing tracer gas into the evolved mixture,
- (d) a pressure control valve in the second line for regulating the pressure of the tracer gas,
- (e) a flow meter in the second line for continuously measuring the rate of flow of the tracer gas, 5
- (f) a flow control valve in the second line,
- (g) a flow-regulating controller connected to the flow-meter and the flow control valve for regulating the flow control valve in accordance with the flow meter indication and for continuously generating signals indicative of the instantaneous rate of flow of tracer gas, 10
- (h) a third pipe line communicating with the first and second lines and through which flows the tracer gas and evolved gas mixture in a sample stream. 15
- (i) a pressure control valve, a first vacuum pump, a pair of gas analyzers, and a second vacuum pump, in the third line in series in the order named, and in the direction of flow of the sample stream, arranged to maintain a substantially constant sample stream flow through and pressure in the analyzers, one of the analyzers being adapted to continuously generate signals indicative of the instantaneous fraction of tracer gas in the sample stream, the other 25

- of the analyzers being adapted to continuously generate signals indicative of the instantaneous fraction of carbon monoxide in the sample stream,
- (j) means for combining all the generated signals to continuously produce an output signal indicative of the instantaneous weight rate of flow of carbon monoxide, and
- (k) means for totalizing the output signals.

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