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CALIBRATION OF GAS CHROMATOGRAPHS FOR IMPROVED PERFORMANCE

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Calibration of Gas Chromatographs for Improved Performance

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Introduction

The Central Area Transmission System (CATS) is a natural gas gathering system located in the U.K. sector of the North Sea. The system is operated by Amoco (U.K.) Exploration Company on behalf of the CATS management group, which besides Amoco includes British Gas, Amerada, Phillips, Fina and Agip. The pipeline is designed to transport 1,600 million standard cubic feet a day of gas through 250 miles (400 km) of 36" diameter pipe. The system pressure varies from 170 bar offshore to the Teesside onshore terminal delivery pressure of 100 bar. The elevated system pressure allows CATS pipeline to accept "rich" natural gas with up to 0.5% Hexanes and heavier hydrocarbons. The gas is delivered to the onshore terminal at Teeside in the dense phase, above the cricondenbar, without the formation of retrograde condensation.

All CATS fiscal measurement for delivery and allocation is based on a component mass-energy using concentric orifice meters (in accordance with ISO 5167), dual on-line gas chromatography and gas density continuously computed using the American Gas Association Report Number 8 (AGA 8) Detail Characterization Method Equation of State. Energy determination is in accordance with ISO 6976 Natural Gas - Calculation of calorific value, density and relative density.

Economic Impact

The economic impact of gas analyses when used to calculate the component mass-energy for commercial purposes is well documented and generally understood within the gas industry. It will suffice to say that the chromatographic analyses yield data that represents more than 50% of the information needed for a total energy calculation. The CATS customary unit for natural gas custody transfer:

Σ (Component tonnes x Gigajoule/tonne) = Total Gigajoules

Principles Of Gas Chromatograph Operation

The elementary principles of the gas chromatograph (GC) are relatively unsophisticated and have been known for many years. The principle is that an individual component will cause a unique response in one of several types of detectors when its presence is sensed. The problem is to separate the individual components in a mixture of natural gas into specific definable component. The evolution of separation media and techniques and detection hardware and methods have resulted in the commercial success of the GC.

Today's GC employs a high purity inert gas which is continually flowed through the instrument. The inert gas most commonly used is helium, but hydrogen or argon can be used when the gas sample contains helium. This gas is referred to as the carrier gas. The carrier gas pressure is carefully regulated. the reduced pressure carrier gas is then filtered to remove moisture. To achieve accurate measurement of the component concentration, an extremely pure carrier gas is mandatory. The recommended carrier gas is an analytical grade or GC grade which is 99.999 percent pure.

The carrier gas is normally flowed through a multi-port sample injection valve before it enters the GC at the heated sample injector. The sample injector valve will be discussed along with the sample injection procedure. The heated sample injector is connected to the chromatograph column (or group of columns) which is used to separate the components in the mixture One way the injector can be used is to inject a sample using a syringe. Syringes are available that can inject a high vapor pressure sample or a liquid. The syringe is inserted into the injector through an external end piece known as the septum.

Required separation is accomplished with one or more columns. Traditional columns are generally made of one-sixteenth to three-sixteenth inch diameter stainless steel tubing. Initially, the tubing is thoroughly cleaned. The column is next packed as tightly as possible with a material that will separate a mixture into individual components. The columns may contain a variety of materials depending on what component separations are required. Column packing generally falls into two groups: "gas-solid" and "gas-liquid". The "gas-solid" column separates the components in the gas using only the surface material and/or the pore structure for the separation. The "gas-liquid" column uses a stationary liquid phase supported by diatomaceous earth, such as Chromosorb P. The newest type of widely used columns utilize capillary tubing normally made of glass. The inside surface of the capillary tubing is coated with a separating media to cause the separation. The capillary tubing is typically 40 to 100 meters long in a laboratory GC. A typical packed column used in a laboratory GC does not exceed 10 meters. The packed columns can not totally separate components heavier than normal pentane. Instead several components are grouped together in the heavier hydrocarbon peaks. The advantage of the capillary column is that heavier hydrocarbon components can be separated allowing cost effective analysis of products as heavy as C_{12}. Hydrocarbons heavier than C_{12} can be accurately analyzed but the time required makes routine gas analysis impractical. The scope of this paper does not allow full discussion of all of the column packing and capillary coating materials; but, sufficient to say, each material used has advantages and disadvantages for various types of separations. Traditional on stream GCs use packed column technology.

GC's are configured with one or more types of detectors to accomplish accurate analyses. The two most commonly chosen detectors are the thermal conductivity and the flame ionization detectors. Seivers, electron capture, and flame photometric are examples of detectors occasionally installed for specific applications. The most specific detector employed is the mass spectrometer. This relatively expensive detector is used to identify

the elution order of the various components in a gas mixture containing a variety of heavier hydrocarbon components. The thermal conductive detector (TCD) is a special resistor that responds to specific compounds based on thermal conductivity with a linearity of 10^4. The flame ionization detector (FID) uses the principle that the electrical conductivity of a gas is directly proportional to the concentration of charged particles within the gas. The effluent gas from the column is channeled through an electrode gap and the organic impurities are ionized by a hydrogen flame. The charged particles within the electrode gap allows a current to flow through the gas and a measuring resistor. The voltage drop is recorded and integrated. The major advantage of this detector is its high sensitivity to organic compounds, greater than 10^6. The disadvantage is the detector gives little or no response to the following components:

He	Ar	$SiHCl_3$	Ne
Xe	O_2	N_2	CS_2
COS	SiF_4	H_2S	SO_2
N_2O	NO_2	NH_3	CO
CO_2	H_2O	$SiCl_4$	

On stream GCs normally use TCD technology. The two commonly used systems are filament or hot wire and thermister. The hot wire is a thin section of wire installed in the detector. The hot wire is operated in a carrier gas atmosphere. The carrier gas serves two purposes: provide a base thermoconductivity so that an expected detector response can be determined for use as a base line and to cool the filament. Without carrier gas flow the powered filament will be damaged. The thermister is more rugged and less susceptible to damage when the carrier gas runs out. Two carrier gas cylinders with an automatic switch over feature are recommended. The operator must monitor the carrier gas pressure so that carrier gas replacement is done on a timely basis.

The gas sample is injected into the carrier gas using a gas injection valve. The sample injection valve is normally equipped with an external sample loop. The sample loop ranges in size from approximately 0.25 to 2.00 cc. The smallest sample loop that can produce acceptable results should be used. This will result in the development of maximum linearity for each component analyzed and as well as being able to repeatably detect all of the components in the mixture. The sample injection valve is a four, six, eight or ten port valve that allows the uncontaminated carrier gas to flow through using two ports while the valve is in the normal position. Sample gas is delivered to the sample loop connected to two ports adjacent to the carrier gas ports that can be connected to the flowing carrier gas by rotating the valve. For the most repeatable results, the sample loop is evacuated and then filled with the gas sample to zero gauge. An alternate method used by most on stream GC manufacturers is to purge the sample loop and reduce the sample loop pressure to near zero before the gas sample is injected. It may be necessary to insulate and heat trace the sample injection valve and the connecting tubing to the injector.

GC Calibration Standard

The standards must be heated to 60° C for an adequate period of time to assure that all of the heavier components are forced into the gaseous phase and adequate mixing has occurred. A 5° C differential temperature between the bottom and top of the sample cylinder is sufficient to mix the sample. For optimum performance, the standard sample cylinders should be heated all of the time which allows use whenever desired. The standard gas samples should also be blended to produce the desired heating value and relative density as well as the required compositional analyses. Natural gas standards should be replaced when the pressure falls below 3-4 barg or 50 percent of the pressure achieved after blending is depleted, whichever is lower, or when the standard is one year old. If there are significant gas composition fluctuations in the flowing stream causing the relative density changes greater than +/- 2%, due to economic reasons, variations in cold plant operations or changes in producing zones or wells, two or more calibration standards may be required.

An excellent check of the standard is accomplished by sending the standards to a responsible third party laboratory that can perform an extended analysis. If the standard vendors analysis and the third party laboratory analysis agree within the tolerances expected, the standard is deemed satisfactory. The acceptable GC tolerances for the various components have been reduced commensurate with Amoco's extensive test matrix.

Composition	Repeatability	Reproducibility
N_2	2.0 %	4.0 %
CO_2	2.0 %	4.0 %
C_1	0.2 %	0.7 %
C_2	1.0 %	2.0 %
C_3	1.0 %	2.0 %
IC_4	2.0 %	4.0 %
NC_4	2.0 %	4.0 %
IC_5	2.0 %	4.0 %
NC_5	2.0 %	4.0 %
C_6 GROUP	5.0 %	10.0 %
C_7+	5.0 %	10.0 %

CATS Operator procedures involve sending the new calibration gas standard to the designated third party laboratory (used for monthly composite sample extended analysis for C6+ or C7+ characterization) to cross correlate and verify the sample and the lab simultaneously. If agreement is not achieved per the table above, the sample should be returned to the vendor for replacement or the lab methods and procedures should be verified. Another method of quickly verifying that a new standard is satisfactory is to acquire and test the new standard on your GC as an unknown gas before the existing standard is depleted below a satisfactory pressure or becomes too old.

The separation of the gas mixture is accomplished using the column(s) carrier gas flow rate and oven temperature. The flow rate of the carrier gas ranges between 20 and 60 cc per minute; but, the flow rate usually is about 30 cc per minute. The flow rate should be verified and logged at least daily and when peak elution times begin to drift. The operating pressure is approximately 8.5 barg. The carrier gas cylinder pressure should be checked often and the GC should be automatically powered down if the preset carrier gas pressure can not be maintained. Additional security is obtained when several carrier gas cylinders are connected to the supply system using commercially available mullet cylinder connection equipment.

The column oven temperature is normally operated in one of two modes to aid in component separation:
- isothermal
- temperature programmed

A GC can produce accurate results in either mode but, the efficiency of the heavier hydrocarbon component separation is more limited when operating isothermally. If good lighter component separation is obtained, the heavier peaks may become very broad with a low amplitude which does not contribute to repeatable results. A two plateau temperature program and the proper temperature ramping rate can produce much improved results. A typical temperature program is shown below for illustration purposes; but, each new column(s) installed will exhibit a different efficiency based on liquid phase coating, support media size, and how tight the column was packed.

The temperature program for a DC 200/500 stationary liquid phase on a 80/100 mesh Chromosorb P acid washed base is detailed below. This is the most common single column used to separate hydrocarbons through C_5's along with N_2 and CO_2.

	Column Temperature	Duration	Program Rate
Initial Temperature	70 ° C	2 Minutes	
Ramping Rate			32 ° C per Minute
Final Temperature	180 ° C	9 Minutes	

The detector temperature should be set higher than the maximum oven temperature based on the manufacturers recommendation, usually 25 to 50° C. The injector zone temperature, the oven temperature, and the detector zone temperature should be monitored and logged daily. A failure in the temperature control system will cause inaccurate measurement.

Most on stream GC's operate isothermally and most laboratory GC's can be temperature programmed. Laboratories have been checked that could use temperature programming;

but, choose to continue to test isothermally. Both efficiency and accuracy may be improved with temperature programming.

ISO 6569 Natural Gas - Rapid Analysis by Gas Chromatograph and/or GPA Standard 2261, Analysis of Natural Gas and Similar Gaseous Mixtures by Gas Chromatography should be followed to achieve the best results.

Sampling for On-line Analysis

Samples points for on stream gas chromatographs require special attention to assure proper performance. A sample heating method is required at or before the point that the sample pressure is reduced for the GC. The sample line must also be completely heat traced and insulated up to the point that it enters the GC to avoid heavy hydrocarbon liquid drop out. The temperature maintained in the sample line must be a minimum of 11 to 28 °C above the flowing pipeline temperature to assure the hydrocarbon dew point is never reached. Ideally, the sample line will have a continuous slope from the sample source to the end device so that any free liquid will drain back into the pipeline. Realistically, other equipment and the design of the building may force the sample line to be routed differently. In this case, a free liquid trap should be installed at all low points to allow to assure the GC will operate properly.

Sample Cylinder Preparation

The first item needed to obtain an adequate sample is a clean sample cylinder with both valves in good repair. The valves should be checked using either/or both high pressure and vacuum to assure no leakage is present. The choice of how to test the sample valves depends totally on how the sample cylinder will be used to obtain the sample. A high pressure check is done by immersing the pressurized cylinder and both valves in water. A vacuum pressure test is accomplished by observing a vacuum test gauge or manometer for approximately ten minutes. For a piston cylinder, both sides of the piston should be pressurized for the water test. Standard cylinders should be cleaned using steam to assure they are properly prepared. The proper use of steam cleaning will assure that the heavy hydrocarbons are removed from the sample cylinder. Piston cylinders should be cleaned in accordance with the manufacturers recommendations for the removal of heavy hydrocarbons. All cylinders should be dried using dehydrated air or an inert gas that is free of water. Finally the cylinder is evacuated using a vacuum pump. Cylinders lined with materials like Teflon have been used in a effort to control the loss of H_2S and other reactive compounds.

Specification of Composition for the Calibration Gas Standard

Based on years of experience in chromatography, Amoco/CATS recommends for optimal chromatograph performance, the composition of the gas used for periodic calibration of the GC (cal gas) should include all naturally occurring hydrocarbons in sufficient quantity for adequate peak resolution. An example of the proper calibration gas is described as follows:

COMPONENT	COMPOSITION
2-Methylpentane	0.20 %
2,2-Dimethylbutane	0.05 %
Cyclohexane	0.05 %
N-Heptane	0.30 %
N-Hexane	0.20 %
N- Pentane	0.35 %
Isopentane	0.35 %
Neopentane	0.10 %
N-Butane	1.00 %
Iso-Butane	0.50 %
Propane	3.00 %
Ethane	7.00 %
Carbon Dioxide	2.00 %
Nitrogen	1.50 %
Methane	Balance

Calibration standards should be a gravimetric mixture produced using weights traceable to N.P.L. standards. The certificate of composition data should include the chemist's name, date certified, cylinder size, cylinder serial number, valve type and the actual composition.

This standard composition is a departure from other schools of thought that suggest the use of a similar (to the flowing stream) gas composition. Apparently, for economic reasons some advocates recommend using a spot sample of the actual gas stream (analyzed at a 3rd party laboratory) as the calibration gas standard. Amoco strongly disagrees with this approach for two primary reasons; it does not insure detector linearity over a normal range of variations in gas composition and it adds another step in the laboratory tracability.

The initial Teesside sales gas GC standard was the vendor's recommended, lean standard which did not require additional heat due to it's lower hydrocarbon dew point. After start up and commissioning of the system, a new, heated (60 C) standard with the above composition was installed and both GCs immediately produced a step change in Relative Density of + **0.6** % (approximate change in mass of 0.3%) !

Chromatograph Performance Verification

CATS Operator is required to conduct natural gas chromatograph performance verifications (audits) to assure that laboratory and on stream instruments provide accurate gas analyses for the calculation of calorific value, density and relative density. The procedure guarantees commercial exchanges are made on an equitable basis. Practically speaking many factors affect detector performance requiring two or more samples covering the range of the unknown gases to be analyzed to assure adequate detector performance. A family of highly accurate gas standards is mandatory before any gas chromatograph can be confidently verified. These standards must contain the components that will be present in the unknown sample. If the detectors used in the gas chromatograph were totally linear, verification and calibration could be accomplished with a single, lean natural gas standard. Extrapolating the response factors for the heavy hydrocarbon gases from the experimentally determined response factors for the lighter hydrocarbon factors is extremely dangerous. Naturally occurring heavy hydrocarbons with the same number of carbon atoms do not all have the same thermoconductivity factors. When extrapolated response factors are used, the amount of error introduced in compressibility, relative density, and heating value calculations varies with the amount of heavy hydrocarbons present.

This verification procedure utilizes natural gas samples that represent the range of components that would be normally found in CATS transported natural gas. Once properly heated, the samples are analyzed by each gas chromatograph at the facility. The resulting mole percentages are compared for accuracy, and each chromatograph's relative response factors for normal compounds are used for a plot to check the chromatograph's detector linearity. The test procedures and a critical review of practices and procedures can determine the ability of a chromatograph to provide analyses and resulting calorific value calculations that are repeatable within +/- 0.25 %.

The response factors developed for each standard are used to check the performance of the GC. A procedure for plotting the log of the molecular weight of the each normal component versus the log of the relative response factor of that component is detailed in GPA Standard 2177, Tentative Method for the Analysis of Demethanized Hydrocarbon Liquid Mixtures Containing Nitrogen and Carbon Dioxide by Gas Chromatography. Propane has been chosen as the compound to which all of the other compounds are related. The resulting curve is normally referred to as a "Relative Response Factor (RRF) Plot". If the resultant plot is a straight line, the detector is operating in the linear range.

Calibration Results

Using the recommended calibration gas composition described herein, the two Teeside chromatographs have been calibrated on a weekly basis. Performance evaluations using the above described method (Log - Log plot of RRF vs Mol. Wt.) are provided in Figures 1-24. Each graph includes two runs each from both machines, or in other words each plot

represents four lines that essentially line on a single path. In addition to near perfect detector linearity, these results support Amoco's reasons for using a rich cal gas by establishing exceptional variability between the two machines as the difference in computed Molecular Weight is typically less than 0.02 %.

A statistical evaluation (standard deviation and 95% confidence values) of the average Relative Response Factors from these calibrations is shown below:

Teeside GC Analyzer Number 1

Composition	Avg. RRF	Std. Deviation	95 % Confidence
Methane	2.035515	0.010406	0.004163
Nitrogen	1.695678	0.019662	0.007866
Ethane	1.271405	0.004171	0.001669
Neo-Pentane	0.804509	0.003452	0.001381
Carbon Dioxide	1.397482	0.006560	0.002624
Propane	1.000000	0.000000	0.000000
Iso-Butane	0.846650	0.001466	0.000587
Normal Butane	0.826140	0.001834	0.000743
Iso-Pentane	0.733816	0.004412	0.001765
Normal Pentane	0.713262	0.005790	0.002316
Hexane Group	0.648190	0.011149	0.004461
Heptanes Plus	0.525399	0.013029	0.005212

Teeside GC Analyzer Number 2

Composition	Avg. RRF	Std. Deviation	95 % Confidence
Methane	2.008504	0.008850	0.003617
Nitrogen	1.660023	0.018327	0.007490
Ethane	1.263057	0.004235	0.001731
Neo-Pentane	0.768648	0.146725	0.059963
Carbon Dioxide	1.380554	0.005987	0.002447
Propane	1.000000	0.000000	0.000000
Iso-Butane	0.844928	0.001391	0.000568
Normal Butane	0.826427	0.001563	0.000639
Iso-Pentane	0.737460	0.003470	0.001418
Normal Pentane	0.716241	0.004470	0.001827
Hexane Group	0.649519	0.009219	0.003768
Heptanes Plus	0.519532	0.014650	0.005987

Worldwide GC Survey Results

Initial Survey Results

Within the last three years, thirty-six locations have been evaluated by using the previously outlined method. Nine of those locations have been revisited since the initial survey. A copy of the survey form is attached. It should be noted that only six of the initial thirty-six laboratories surveyed were determined to have no discrepancies. Nine of the laboratories were left with no discrepancies as a result of the cooperative efforts of the Amoco survey team and personnel of the companies being surveyed. This cooperation resulted in "point forward" accurate analyses. The discrepancies reported were placed into seven categories. These are:

- Not heating field samples
- Non linear response factors
- Calibration gas not properly heated
- Calibration standard out of date
- Primary calibration gas not used
- Integration, calculation or recognition of peaks
- Calibration standard not representative

The discrepancy that occurred most frequently was "calibration gas not being heated". "Primary calibration gas not used " only occurred once.

Follow-up (Return) Results

Of primary interest is the additional category, "Other". This category was added to reflect the new discrepancies that were noted at the return visit but were not noted during the initial survey. Two of the eight labs revisited were found to have corrected all initial discrepancies. The highest occurring discrepancy was once again, "calibration gas not properly heated", although this number was reduced from the initial occurrences.

Conclusions

The use of a proper composition calibration standard, heated to insure there is no separation due to gravity segregation is economical and necessary for accurate gas measurement on an energy basis.

Properly designed and calibrated gas chromatographs demonstrate optimal detector linearity with minimal variability between machines.

The survey method describe herein has been extensively used over the last two years to evaluate laboratory and "on-line" gas chromatograph performance in the United States, the United Kingdom, Norway and Trinidad. More than twenty million dollars in lost revenue have been identified as a result of these tests.

The major benefits of this approach to GC performance evaluation include:
- Immediate trouble shooting and evaluation of performance
- Immediate elimination of errors on a "point forward" basis in many cases
- Elimination of return trips for follow up testing
- Partnering with other companies to achieve a common quality assurance
- Continuity

In the future, only the companies that assure accurate measurement in an efficient manner will survive.
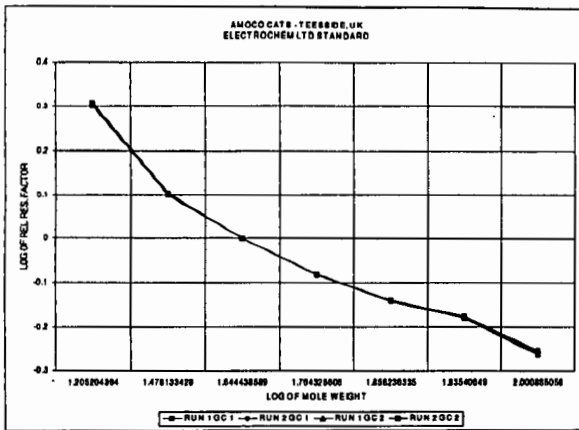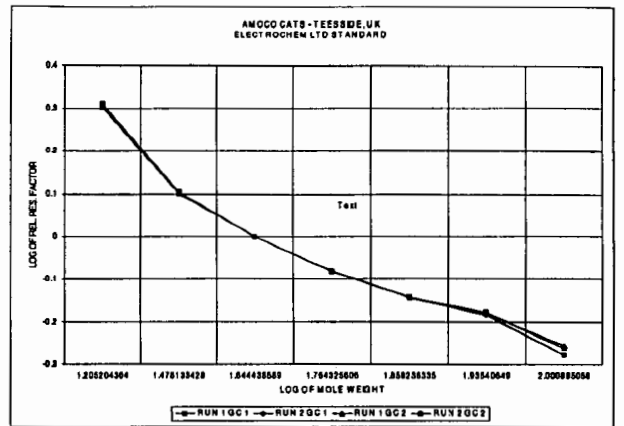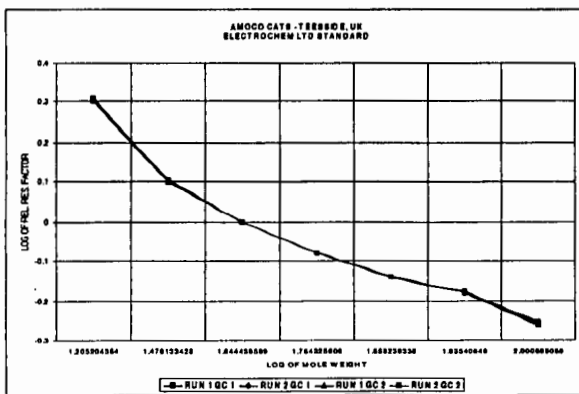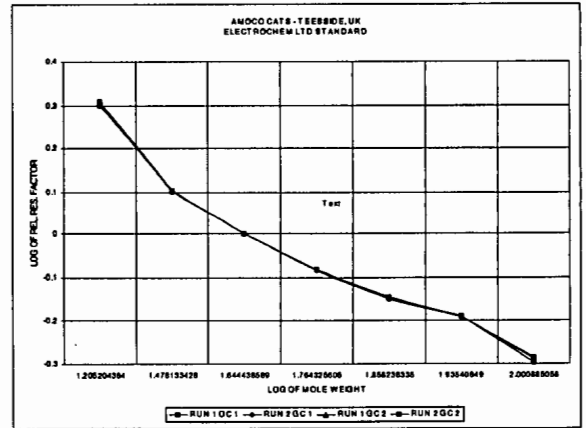
Figure 1

Figure 2

Figure 3
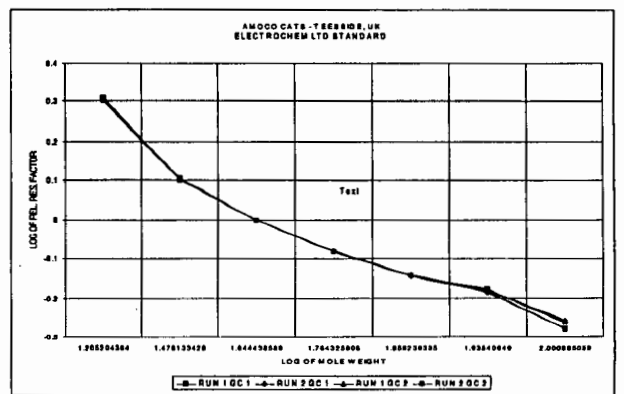
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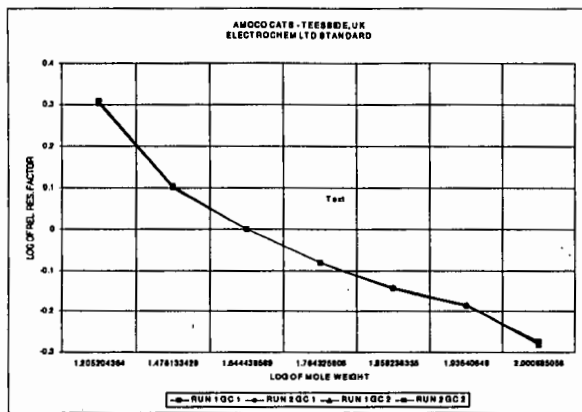
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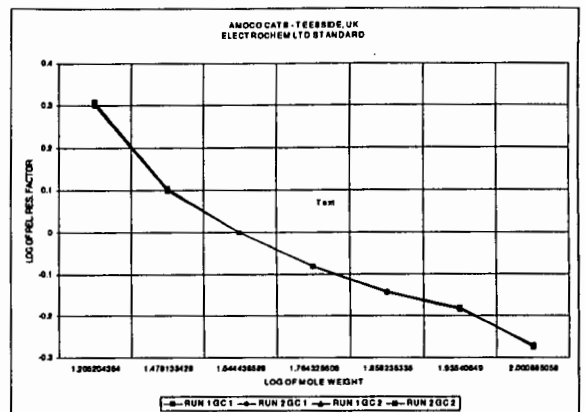
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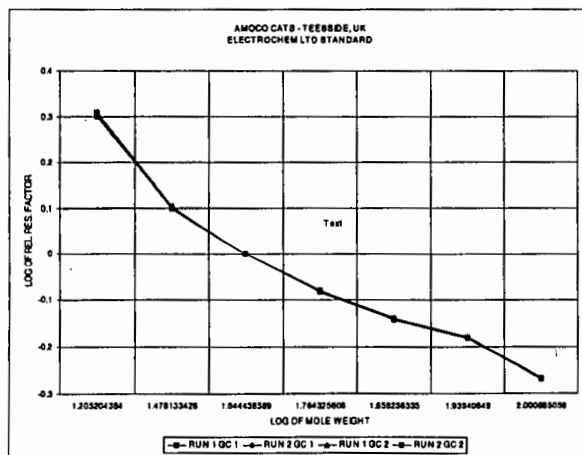
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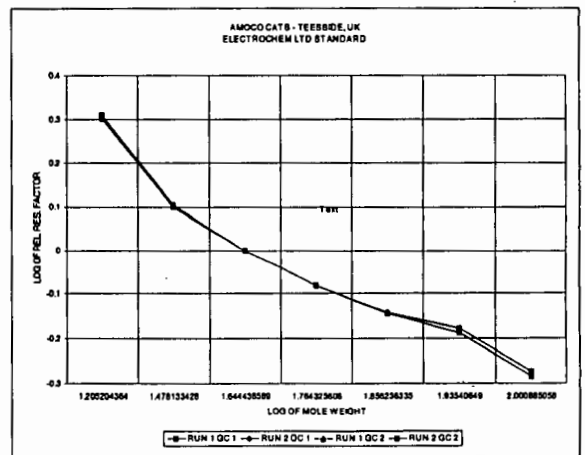
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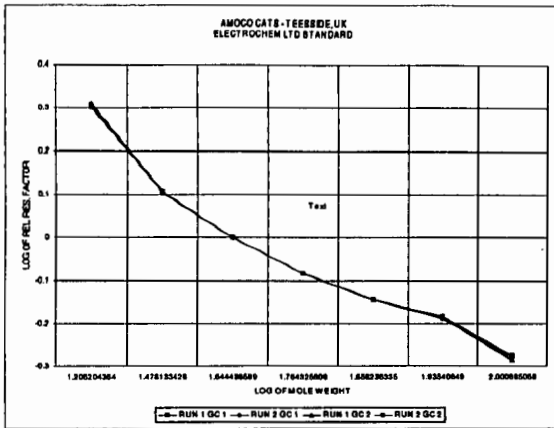
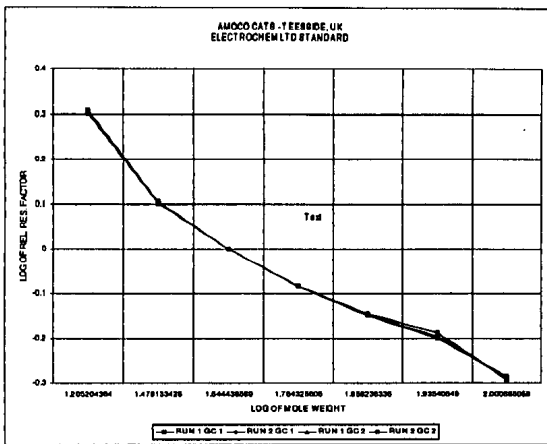Figure 9
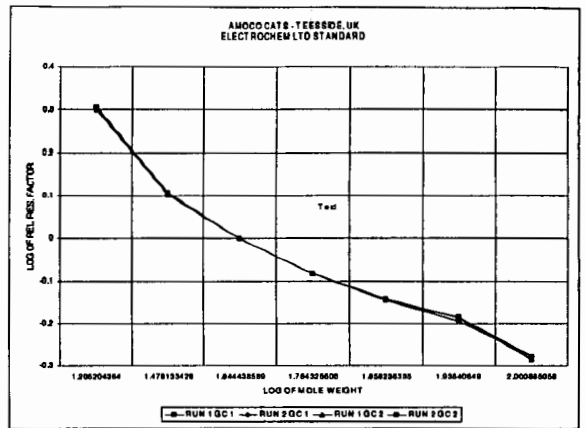
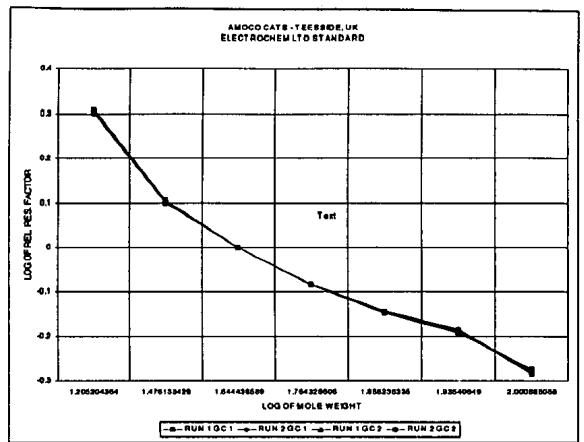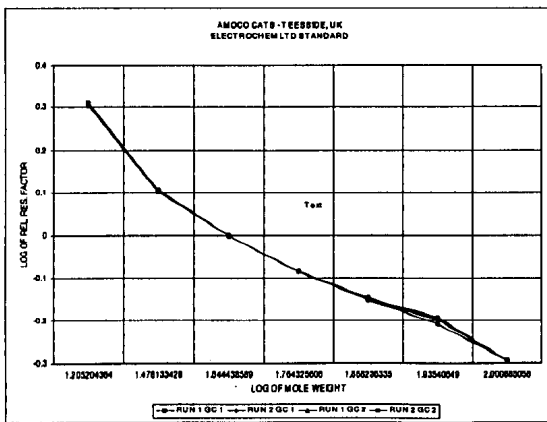Figure 10
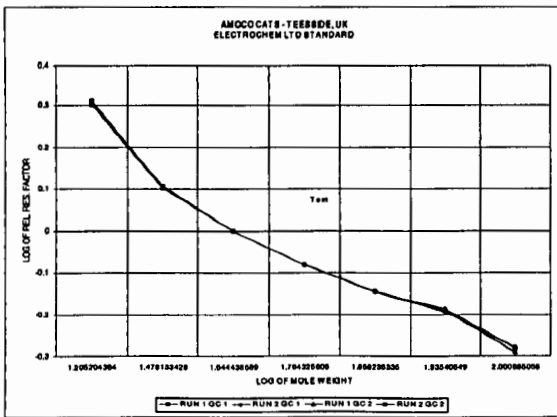
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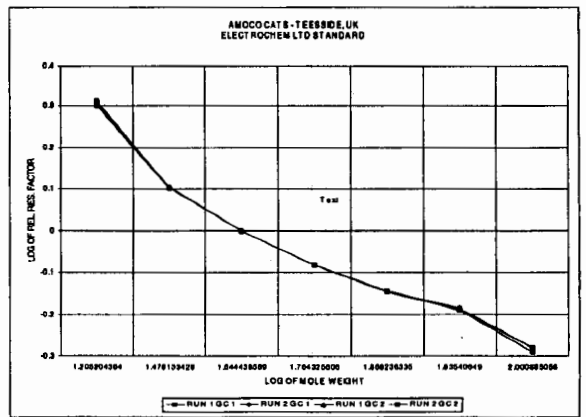
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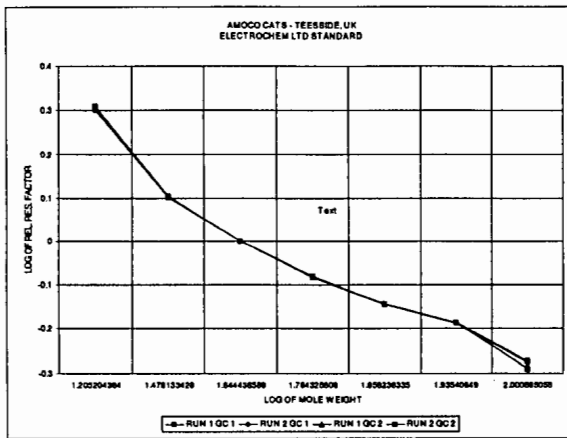
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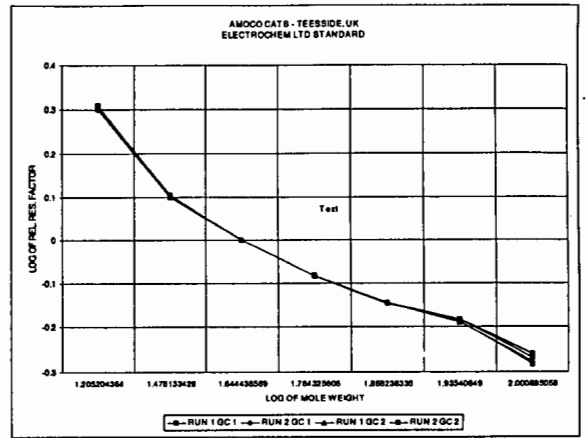
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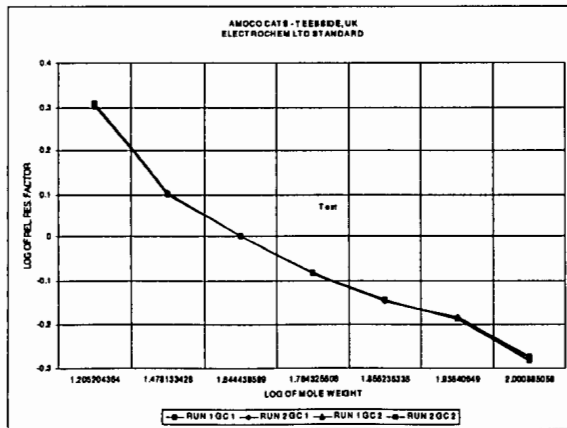
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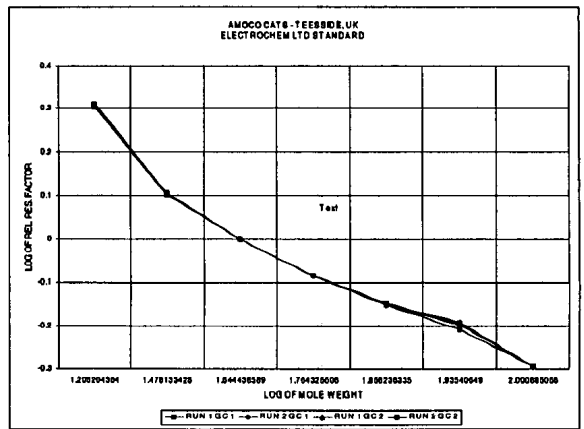

Figure 16

Figure 17

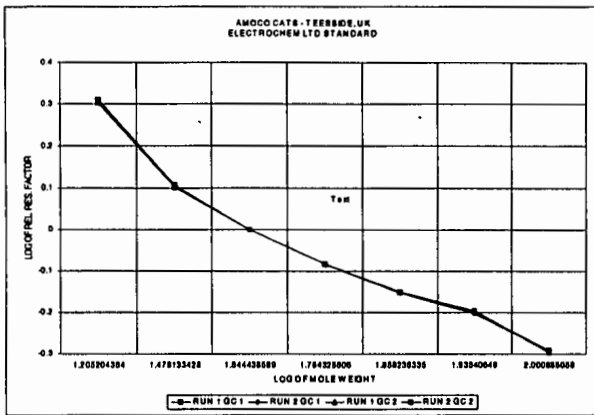**Figure 18**

Figure 19

Figure 20

Figure 21

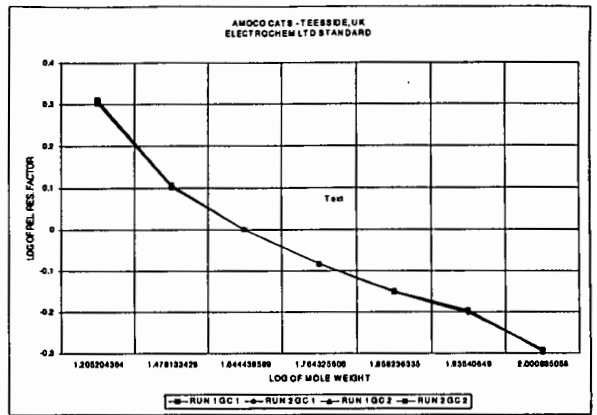

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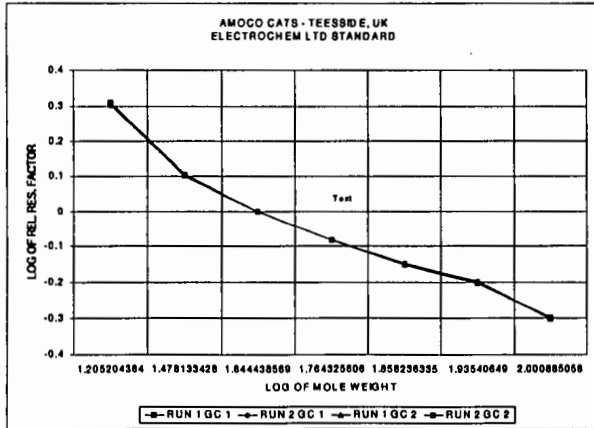
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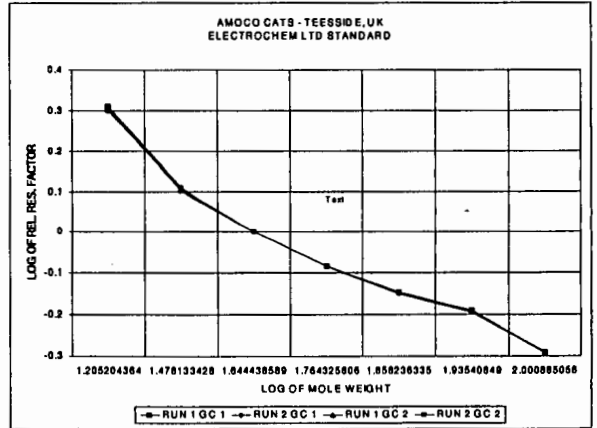
Figure 24

PROVIDE THE FOLLOWING EQUIPMENT DETAIL FOR EACH ANALYZER TO BE USED:

:   **Analyzer**

Make   _____

Model   _____

Column Configuration

_____

(Column and Description)

_____

                            _____

                            _____

                            _____

Type of Detectors   _____

          Integrator

Make   _____

Model   _____

Last Component Analyzed

(i.e. C_5, C_6, C_6+)   _____

TEAM CHECK LIST

1. **Sample Handling & Conditioning**

	Yes	No
Samples Heated	—	—
Temperature Monitored	—	—
Temperature at 140 °F (+/- 5 °F)	—	—
Time of sample heating monitored	—	—
Duration of heating in hours		
NOTE: Normal heating time is 24 hr (± 12 hr)		
Analyzer room heated	—	—
Analyzer room Air-conditioned	—	—
Sample taken immediately from heater to analyzer if manually transferred	—	—
Are connections, lines & hardware between sample Cylinder and Analyzer insulated	—	—
Are connections, lines & hardware between sample Cylinder and Analyzer heated	—	—
Sample loop size is		
0.25 cc	—	—
0.50 cc	—	—
1.00 cc	—	—
Other (Specify size)		
Vacuum injection system	—	—
Purge injection system	—	—
If a purge system is used, is there back pressure	—	—
Is the purge rate read or measured	—	—
Record the purge rate		
Helium carrier gas is used	—	—
Is carrier gas pressure monitored	—	—
Is the carrier gas flow rate monitored	—	—
If yes, record the flow rate in cc/minute		
Carrier gas is 99.999 purity	—	—
If no, record the carrier gas purity used		

2. Analyzer

	Yes	No
Is this an isothermal run	—	—
If yes, record Temperature in °C		.
NOTE: If no, secure copy of temperature program		
Are the columns configured per GPA 2261	—	—
If no, list the configuration		
Integration method is:		
Peak Height	—	—
Area	—	—
Highest carbon number component analyzed is:		
C6	—	—
C6+	—	—
C7	—	—
C7+	—	—
Other (Specify)		
NOTE: Team secure Chromatogram		
Calibration schedule is		
Daily	—	—
Weekly	—	—
Monthly	—	—
Other (Specify)		
Analysis frequency is:		
Daily	—	—
Weekly	—	—
Monthly	—	—
Other (specify)		

CALIBRATION STANDARD GAS

	Yes	No
Manufacturer		
Less than one year old	—	—
If no, list the date blended		
NOTE: Team secure a copy of analysis of the standards normally used.		
Is the standard heated continuously	—	—
If no, list the length of time heated		
Is standard heated to 140 °F (± 5 °F)	—	—
If no, state the temperature in °F		
Is an insulation blanket used	—	—
Is pressure on the standard monitored	—	—
If yes, record the pressure in PSIG		
Is the Dew Pt. provided		
If yes, record Dew Point		
Is sample ever exposed to Temp. below Dew Point	—	—

CALCULATION

Yes No

Performed in accordance with the latest 2145 (1994) ___

___ Performed in accordance with the latest 2172 (1994) ___

___ Other methods used ___ ___
      If other, please specify

_____ Values for C6+ or other heavy Fraction
      C_6 ___ ___
      C_{6+} ___ ___
      C_7 ___ ___
      C_{7+} ___ ___
      Other (Specify)

_____ Composition of Fraction
      C_6 ___ ___
      C_7 ___ ___
      C_{8+} ___ ___
      Other (Specify)

_____ Resolution, number of significant figures

_____ Control Charts used ___ ___
      Other (State) _____
Long term logging of response factors ___

___

NOTE: Rating by Team

_____

References

[1] Paper presented at the North Sea Flow Measurement Workshop, a workshop arranged by NFOGM & TUV-NEL

Note that this reference was not part of the original paper, but has been added subsequently to make the paper searchable in Google Scholar.