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**PHOTOMETRIC ON-LINE MONITOR
FOR TOTAL OXIDANTS
IN THE PORTS PURGE CASCADE**

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CONTENTS

LIST OF FIGURES	v
LIST OF TABLES	v
SUMMARY	vii
INTRODUCTION	1
APPARATUS AND PROCEDURE	3
RESULTS AND DISCUSSION	5
SALT REACTOR METHOD	7
CALCULATIONS	9
INTERFERENCES	11
DIRECT METHOD	11
CONCLUSIONS AND PROJECTIONS	12

LIST OF FIGURES

1	UV Absorption Spectra of UF_6 , ClF_3 , F_2 , and Cl_2	2
2	Gas System Schematic	4
3	Reactor Vessels	6
4	Beer's Law Plots	8

LIST OF TABLES

1	Calibration data for F_2 , ClF_3 , and Cl_2 in N_2	9
2	Pooled data for F_2 , ClF_3 , and mixed $\text{F}_2 + \text{ClF}_3$ in N_2	13

SUMMARY

A proposed scheme for the continuous, on-line monitoring of the total concentration of oxidizing (fluorinating) gases in the top purge section of a gaseous diffusion cascade was evaluated using simulated process conditions in a laboratory. The scheme involves the reaction of the oxidizing gases in a sample stream with sodium chloride to produce chlorine, which is then measured with an ultraviolet photometer to yield a value for total oxidant concentration. This report describes the experimental apparatus used in studying this method and presents the resulting data and conclusions. The study found that the technique would work under some conditions, but would be troublesome and probably undependable if implemented as a real-time process monitor. During the study, it was discovered that the photometric approach could provide a measurement of total oxidant concentration without resorting to the salt reaction.

INTRODUCTION

During the course of this investigation, the project was designated a PLRTS instrumentation site milestone for FY 1990. This report is presented in fulfillment of that milestone.

The desired result of this investigation was an analysis for the total concentration of two compounds taken together: Fluorine (F_2) and chlorine trifluoride (ClF_3). These powerful oxidizing (fluorinating) gases may occur in the Portsmouth purge cascade, in mixtures with the other purge gases, including the R-114 process coolant, giving a remote possibility that explosive proportions might occur at some point in the cascade. A direct and continuous "total oxidant" analysis would add further protection against this possibility.

On-line monitoring is a natural setting for photometric analysis. Consequently, photometric analyzers are widely used and commercially available. Generally, the analyzers measure the absorption of radiation at a fixed wavelength where the component of interest absorbs strongly and interferences due to overlapping spectra of other components of the stream are at an insignificant level. These compounds have typical ultraviolet (UV) spectra,^{1,2,3,4} characterized by broad absorption bands, as shown in Fig. 1. The spectrum of F_2 exhibits a relatively weak, broad absorption maximum at 285 nm, while ClF_3 , a stronger absorber, exhibits no well-defined maximum. Each will interfere in the photometric determination of the other in binary mixtures, to say nothing of interferences from the other components of the purge gas. The figure makes apparent the formidable interference of UF_6 throughout the UV region, except for a minimum at 340 nm.²

Sometimes interferences can be avoided by chemically modifying the analyte species so that their absorption maxima fall in a relatively unchallenged spectral region. A common practice in the photometric analysis of solutions is to add reagents that react with the analytes to yield products whose spectra differ in a desired way, e.g., increased sensitivity or absorption maxima at wavelengths different from those of the interferences. An analogous procedure in the case of these powerful oxidizers uses the dry chemical reaction between them and sodium chloride. When a gas stream containing F_2 or ClF_3 contacts dry NaCl, the chloride is oxidized to yield gaseous Cl_2 and NaF:

If the reactions can be conducted quantitatively, the chlorine released gives a measure of the total concentration of both reactants. The Cl_2 product is a stronger UV absorber than F_2 or ClF_3 , so the sensitivity of the measurement should increase. And, since the chlorine absorption maximum falls in the "notch" of the UF_6 absorption spectrum, the treatment achieves not only greater sensitivity but also a shift of the adsorbance maximum to a more desirable neighborhood. This "salt reactor" concept has been evaluated in the laboratory, and the apparatus that has been assembled could be useful in a mobile assembly for further evaluation in the purge cascade.

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(U) Fig. 1. UV Absorption Spectra of UF_6 , ClF_3 , F_2 , and Cl_2 .

APPARATUS AND PROCEDURE

The DuPont Model 400 photometric analyzer was chosen for this work, partly because it is a sensitive and rugged instrument, and partly because we had one on hand at the outset. The 400 analyzer splits the radiation beam from a UV source after it has passed through the absorption cell and directs the two resulting beams through filters which isolate an analytical band and a reference band. The intensities of the analytical and reference bands are measured and converted to absorbance values by the photometric circuit. The split beam technique helps correct for variations in source intensity, dirty cell windows, and some constant interferences in the sample stream since both sample and reference beams are exposed identically to these effects.

Two analyzers were needed to measure the absorbances before and after the reaction with NaCl. We had an analyzer already mounted on a sturdy sampling buggy from a previous project for the K-25 purge cascade, and another analyzer was acquired from standby equipment storage to provide the second channel. Both were upgraded with new source lamps and new filters for various wavelengths. The filters used for the measuring beams had about 25% transmittance at the nominal peak with a bandwidth of 10 nm (full width at half height). The filters for the reference beams had a bandwidth of 50 nm. A reference wavelength well into the visible spectrum was chosen in order to avoid the "tails" of the halogen spectra which extend into the visible blue region. (These tails impart the characteristic pale greenish-yellow color to fluorine, and the green to chlorine gas.) The full-scale absorbance of both photometers was arbitrarily set equal to one absorbance unit. The absorption cell of the inlet photometer had a pathlength of 50.8 cm, and the outlet cell's pathlength was 6.15 cm.

The gas-handling apparatus assembled for this research is shown schematically in Fig. 2. It comprised basically three sections that were interconnected by flexible Teflon tubing. The first section consisted of a manifold with numerous connections enabling introduction of various gases. It included a mechanical vacuum pump and a large soda lime and alumina chemical trap which was normally kept evacuated to provide an emergency dump of the system gas (so far no such need has arisen). The second section included a 69-liter MD cylinder, a Bourdon pressure indicator, and a Metal Bellows pump connected with tubes and valves to allow mixing the contents of the cylinder by circulation, either in a recycle mode or through a loop including the analyzer cells. This was used to premix ClF_3 and N_2 for feeding into the third (dynamic mixing) section, since undiluted ClF_3 is troublesome to blend reproducibly into the sample stream. The third section was a manifold in which accurately measured flows of as many as four gases could be admitted independently at controlled flow rates into a mixing tube. The flow rates were monitored with Hastings type NALL mass flowmeters having Monel transducers. The total flow exiting the mixing tube was also measured to allow calculation of the concentrations thus generated. The mixture flowed from this section through the photometric cells to a chemical trap. The pressure in the cells, monitored by an MKS Baratron type 170M pressure indicator, was adjusted by manipulation of parallel coarse and fine control valves at the outlet from the mixing manifold and the inlet to the final trap. Flow was maintained by continuous evacuation of the trap, either by a Welch roughing pump or a Metal Bellows pump. The Metal Bellows pump was part of

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Fig. 2. Gas System Schematic.

another assembly with an MD cylinder like that shown in Fig. 2. The large cylinder attenuated the substantial pressure pulses created by the pump.

All observations were made at a nominal flow rate of one standard liter per minute (slpm). Sample streams were blended at room temperature by injecting F_2 , ClF_3 , Cl_2 , and N_2 into the mixing manifold and preheated by a short, heated tube before entering the absorption cells which were thermostatted at $180^\circ F$.

The output signals from the photometers, the flowmeters, and the Baratron were connected to an XT-type computer via a Keithley System 570 Data Acquisition Workstation. It was necessary to modify the photometer output circuits to obtain a 5-volt signal suitable for the Keithley interface. The computer was programmed to collect readings from the seven channels, apply a correction for the particular gas or mixture to each flowmeter reading, and display the results on the screen at 2-s intervals. The flow rates were shown as fractions of the total flow. With the readouts of all channels appearing every two seconds, it was relatively easy for the operator to obtain the desired composition, total flow, and total pressure of the sample stream by manipulating the valves while observing the display. When the displayed values appeared stable, a keystroke would initiate collection and storage of (typically) three groups of ten data sets. The average and standard deviation of each element of the ten sets were also computed and stored. Thus, each value given in Tables 1 and 2 is the average of not less than ten single observations.

Two reactor vessels (Fig. 3) were used. The smaller vessel (right) was made from a 5/8-in-diam copper tube with high-vacuum fittings brazed to the ends. Fine nickel screens sandwiched between two Teflon gaskets at each end contained the salt. The capacity was about 48 g NaCl. The larger reactor was made from a flanged (Conflat) vacuum fitting, 5 in long x 1.5 in OD. Again, the salt was kept in place by screens at each end. This vessel held about 150 g of NaCl.

RESULTS AND DISCUSSION

The relationship between absorbance and partial pressure for F_2 , ClF_3 , and Cl_2 mixed with N_2 was shown to be insensitive to the total pressure of the sample up to about 300 torr (6 psia). This is important for practical use since it eliminates the need for strict pressure control in a sampling sidestream whose inlet and outlet pressures may vary. Although the concentration ranges of the calibrations were limited by the sizes of the absorption cells chosen and other apparatus parameters, the responses at other concentrations and pathlengths can be predicted. For example, using a 1-m pathlength would approximately double the absorbance observed in the 50.8 cm cell, thus extending the detection limit to lower concentrations. Similarly, the upper limit could be increased by using shorter cells or by resetting the analyzer's absorbance range. The latter technique has practical limits because although the full scale range of these photometers can be arbitrarily set, nonlinearity usually becomes apparent beyond 1.5 absorbance units.

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Fig. 3. Reactor Vessels

Beer's law gives the relationship between absorbance, A , and the concentration of an absorbing species:

$$A = alc \quad (3)$$

where l is the pathlength in cm and c is the concentration in moles per liter. The molar absorptivity, a , is commonly reported in the literature as the measure of the strength of an absorber. The molar absorptivities obtained in this research for Cl_2 , ClF_3 , and F_2 compared favorably with those available from the literature.^{3,4} This gives some confidence, although such comparisons should probably be considered order-of-magnitude estimates, since the reported values are influenced by the type of instrument used, among other parameters.

Representative calibration curves obtained in this investigation are shown in Fig. 4, and the data used to construct these curves are given in Table 1. Only one line is shown in the figure for F_2 and ClF_3 because the regression lines are indistinguishable on this scale. The data are presented as partial pressure calculated from the flow and total pressure readings (taken), and partial pressure estimated from the regression calculation (found), and the difference, D , expressed as percent of the pressure taken.

The data typically fall within 10% of the line, with occasional outlying values exceeding justifiable rejection limits, such as the point shown in the ClF_3 column for .4 torr taken.

SALT REACTOR METHOD

Nearly stoichiometric evolution of Cl_2 from NaCl by F_2 and ClF_3 individually and collectively, flowing at 1 slpm through the 150-g reactor, was demonstrated. Recoveries of 90-92% were found by measuring the product Cl_2 absorbance when the oxidants were delivered into the reactor at a total concentration of about 5%. Examination of the crystals removed from the reactor showed that the reaction moved forward in a well-defined front. A subsequent experiment in which the product absorbance was measured as a function of integrated flow showed that the breakthrough when the salt is consumed is quantitative and distinct. Thus, a usable lifetime for a given quantity of salt can be predicted if a good estimate of the anticipated exposure level is available. Practically, it would be necessary to establish a reasonable frequency for reactor refurbishment based on experience with real process exposure levels. It should be noted that, given the current regulatory climate, the NaF generated would not only constitute a disposal problem in its own right, but any UF_6 in the stream at that point would be trapped by the NaF .

Reactor overheating may be the most serious drawback for the method, since considerable heat is generated, particularly by the reaction with ClF_3 . The effect was apparent in the small reactor which became uncomfortably warm when 3% ClF_3 in N_2 at 300 torr was fed through it at 1 slpm. The temperature measured at the surface of the copper tube rose from 77 to 150°F and appeared to be reaching equilibrium in 15 min. Later experience with the larger reactor and higher concentrations produced evidence that the heating effect may render the method useless at this flow rate for higher concentrations. When the concentrations were gradually increased to the vicinity of 10%, the surface temperature continued to rise until the experiment was terminated after 25 min. At that time the surface

Fig. 4. Beer's Law Plots

Table 1. Calibration data for F₂, ClF₃, and Cl₂ in N₂

torr F ₂				torr ClF ₃				torr Cl ₂			
A	Taken	Found	D	A	Taken	Found	D	A	Taken	Found	D
0.019	3.1	3.1	-1	0.004	0.4	0.9	139	0.046	2.3	2.1	-7
0.019	3.1	3.1	1	0.007	1.4	1.4	1	0.088	4.4	4.4	-1
0.038	6.3	5.8	-8	0.025	3.8	4.1	7	0.104	5.2	5.2	0
0.040	6.3	6.1	-3	0.025	3.8	4.0	7	0.154	7.8	7.9	2
0.048	7.1	7.2	2	0.025	3.7	4.1	10	0.154	7.9	7.9	1
0.047	7.1	7.2	1	0.032	5.0	5.1	2	0.200	10.3	10.4	1
0.090	13.2	13.2	0	0.032	5.0	5.1	2	0.246	12.8	12.8	0
0.088	13.0	13.0	0	0.032	5.0	5.0	2	0.277	14.4	14.5	1
0.149	21.7	21.8	1	0.072	11.4	11.0	-3	0.297	15.6	15.6	0
0.140	20.4	20.4	0	0.072	11.3	11.0	-3	0.335	17.7	17.6	0
0.082	11.9	12.1	1	0.071	11.3	11.0	-3	0.359	18.8	18.9	1
0.081	11.8	12.0	1	0.100	16.0	15.3	-5	0.400	21.2	21.1	0
0.149	21.5	21.7	1	0.101	16.0	15.3	-4	0.437	23.3	23.1	-1
0.140	20.3	20.4	0	0.100	15.9	15.2	-4	0.477	25.1	25.2	1
0.201	29.1	29.2	0	0.198	29.3	29.8	2	0.455	24.1	24.1	0
0.195	28.4	28.3	0	0.197	29.1	29.6	2				
0.225	32.7	32.6	0	0.195	29.0	29.3	1				
0.208	30.2	30.2	0								
0.225	32.7	32.6	0								

Total pressure: 300 torr

Cell pathlength: For F₂ and ClF₃, 50.8 cm; For Cl₂, 6.15 cm

Temperature: 180°F

Flow rate: 1 slpm

temperature was 184°F and was rising at about 14°/min. Cooling the reactor would be necessary as a minimal precaution for any application in which such concentrations may occur.

CALCULATIONS

In the following discussion, Beer's law is utilized in the more convenient form that uses directly measured quantities rather than concentration and molar absorptivity:

$$A/I = \epsilon p \quad (4)$$

where A = absorbance, l = pathlength (cm), p = pressure (torr), and e is the absorption coefficient. The absorption coefficient, e , and the molar absorptivity, a , are related by

$$e = a/760RT \quad (5)$$

where R is the gas constant and T the absolute temperature.

If the reactions proceed according to equations (1) and (2), the quantity of Cl_2 produced is not a unique function of the total-oxidant input; rather, it depends upon the proportions of the two reactants in the input mixture. For example, the same yield — 3 moles of Cl_2 — would result from reacting either 2 moles of F_2 plus 0.5 mole of ClF_3 , or one mole each of F_2 and ClF_3 . The total oxidant input in the first case was 2.5 moles and in the second, 2 moles. It follows that the salt reactor advantages can only be had at the expense of obtaining a second measurement to remove the ambiguity. At least one of the reactants must be determined independently as well as the combined Cl_2 product in order to obtain the total oxidant concentration. Ideally, when two components of a solution are to be determined, two wavelengths would be found, spaced so that each component would have a wavelength where its absorption was the only important one. Failing this, there is a well-known technique for handling mixtures with overlapping spectra where each contributes, but not strongly, at the maximum of the other.⁴ In the present situation, there is only one practical wavelength region — that of the UF_6 minimum — and there is no place where the absorption due to F_2 predominates. Assuming that no Cl_2 exists in the sample stream prior to the reaction with NaCl and that no F_2 or ClF_3 exists afterwards, when the measurements are made at 334 nm before and after the chemical reaction, Beer's law equations can be written to express these conditions

$$A_i/l_i = e_{\text{F}_2} p_{\text{F}_2} + e_{\text{ClF}_3} p_{\text{ClF}_3} + a_i \quad (6)$$

$$A_o/l_o = e_{\text{Cl}_2} [p_{\text{F}_2} + 2p_{\text{ClF}_3}] + a_o \quad (7)$$

where the subscripts, i and o , refer to the inlet and outlet of the reactor and the constant terms, a_i and a_o , arise from the fact that a typical calibration curve has a nonzero intercept.

Experimentally, the partial pressures are obtained from the flow rates (R) and total pressure as

$$P_j = R_j/R_{\text{tot}} \times P_{\text{tot}} \quad (8)$$

The mole fraction, X_j , given by R_j/R_{tot} , is numerically equal to the flow rate when the total flow is 1 slpm, a condition existing at the inlet by design. Since e_{F_2} and e_{ClF_3} are practically identical, as shown in Fig. 4, they can be represented by e_i , permitting the solution of equation (6), which gives

$$X_i = (A_i/l_i - a_i)/e_i \quad (9)$$

and the total oxidant concentration is

$$C_{\text{tot}} = 100X_i \quad (10)$$

The outlet flow, however, is increased by the two-to-one yield of Cl_2 from ClF_3 , so we have

$$X_o = (R_{\text{F}_2} + 2R_{\text{ClF}_3}) / (R_i + 2R_{\text{ClF}_3}) \quad (11)$$

which is not amenable to solution without an independent measurement of the ClF_3 flowrate. Perhaps the simplest solution to this problem would be to measure both inlet and outlet flow rates, making sure that the pressure and temperature were well controlled. The change in flow rate should equal R_{ClF_3} .

INTERFERENCES

As previously noted, the apparent advantages of the salt reaction are increased sensitivity for the fluorinating compounds and relative insensitivity to UF_6 . In the presence of appreciable UF_6 , F_2 and ClF_3 cannot be measured at all at wavelengths where they absorb strongly, but the UF_6 absorptivity decreases dramatically as the wavelength approaches the vicinity of 340 nm, while the other spectra drop much more slowly. The result is that the absorption coefficients of F_2 and ClF_3 are about equal at 334 nm, and both are greater than that of UF_6 . Essentially, the UF_6 interference adds another term to the absorbance expression for the inlet stream only, since the UF_6 would be trapped by the NaF already generated by the oxidizers. The UF_6 absorptivity is about 40% of that of the oxidizers, so the result should be an overestimation of the oxidizer concentration by about 12 torr or 4 mol% for 10% UF_6 in the worst case situation.

Any ClF present in the sample stream would also interfere. It is a strong absorber in its own right — roughly ten times as strong as the other oxidants¹ — so 1% ClF would look like 10% $\text{F}_2 + \text{ClF}_3$ at the inlet. It would also react with NaCl to yield an excess of the Cl_2 product, so that both inlet and outlet absorbances would be enhanced.

DIRECT METHOD

It was possible to demonstrate smooth conversion of Cl^- to Cl_2 for concentrations up to approximately 5%, but attempts to measure the chlorine product from the reactor at the higher concentrations are most delicately described as unpromising. When the total sample concentrations were increased to the 10% vicinity. There was no apparent correlation between the absorbance of the outlet stream and the Cl_2 expected from the input amounts of F_2 and ClF_3 .

Fortunately, it was observed at the same time that the calibration curves for both F_2 and ClF_3 at 334 nm had nearly the same slope. Subsequent calculations treating both as a single entity, simply by measuring the absorbance of the mixture at 334 nm without resort to the salt treatment, yielded good estimates of the total concentration. Unless the observations were merely experimental artifacts, it appeared that the determination of total oxidant concentration could be performed by a single photometer operating at or near 334 nm. A second series of measurements confirmed the results of the first. The data presented in Table 4 were taken from a series of N_2 dilutions of each of the individual compounds and with mixtures of both. When the data were pooled without regard to whether the solute was

one or two components, the resulting A vs P curve predicted the total concentration very well. To be sure, the sensitivity gained by using the NaCl treatment would be sacrificed, but concentrations as low as 2%, which should suffice for "early warning" purposes can be determined with the 50.8-cm cell.

CONCLUSIONS AND PROJECTIONS

On the basis of the foregoing observations, it appears that the salt reactor concept may work under limited conditions, but that it would be better to avoid the awkward handling and disposal problems it entails. Instead, since the simple measurement of the sample absorbance seems to give good results at this one wavelength, the latter possibility should be explored as a more promising alternative. All of the experimental parameters — wavelength, pathlength, absorbance scale, temperature, pressure, flow rates — could be optimized to obtain the best performance. This will be pursued in FY 1991. In preparation for further investigation, we have upgraded the photometers with new solid state detectors and circuitry.

The present sampling buggy could be adapted for experiments in the plant by the addition of a few more parts. For instance, the Metal Bellows pump needs to be mounted on the buggy in case some help is needed to achieve adequate sample flow on site. Other items to be mounted include a Welch pump, a chemical trap, and cylinders of calibration standards and zero gas. All these things are available in close proximity in the current lab setting, although not in portable form.

The justification for using UV photometry, as opposed to the infrared (IR), has to be that diatomic gases have measurable absorption spectra in the UV, but not in the IR. Only the desire to include F_2 in the measurement makes the UV analysis necessary. A more effective analytical system would combine UV and IR capabilities, for which I suggest an FTIR spectrometer rather than a one-wavelength analyzer. Then the situation vis-a-vis interferences could be evaluated in situ, and in real time. A correction for the total oxidant value obtained by the UV photometer could be obtained by a simultaneous FTIR determination of the UF_6 concentration. If not, the IR analysis would at least indicate the need to move to another sampling location, and determine the ClF_3 and R-114 concentrations as well.

Table 2. Pooled data for F_2 , ClF_3 , and mixed $F_2 + ClF_3$ in N_2 .

A_{obs}	Partial Pressure, torr		
	Taken	Found	D, %
0.004	0.4	0.8	>100
0.003	0.2	0.8	>100
0.001	0.0	0.4	>100
0.007	1.4	1.3	-3
0.019	3.1	3.1	-1
0.019	3.1	3.1	1
0.025	3.8	4.0	5
0.025	3.8	4.0	5
0.025	3.7	4.0	8
0.032	5.0	5.0	-0
0.032	5.0	5.0	0
0.032	5.0	4.9	-0
0.038	6.3	5.8	-7
0.040	6.3	6.1	-2
0.048	7.1	7.3	3
0.047	7.1	7.2	3
0.072	11.4	10.8	-5
0.072	11.3	10.8	-4
0.071	11.3	10.8	-4
0.082	11.9	12.3	3
0.081	11.8	12.2	3
0.090	13.2	13.4	2
0.100	16.0	15.0	-7
0.101	16.0	15.0	-6
0.100	15.9	14.9	-6
0.103	16.3	15.4	-5
0.149	21.7	22.2	2
0.140	20.4	20.8	2
0.149	21.5	22.1	3
0.140	20.3	20.8	2
0.153	23.3	22.8	-2
0.163	25.2	24.1	-4
0.201	29.1	29.8	2
0.195	28.4	28.9	2
0.198	29.3	29.3	0
0.197	29.1	29.1	0
0.195	29.0	28.9	-0
0.199	30.1	29.4	-2
0.225	32.7	33.2	2
0.208	30.2	30.8	2
0.225	32.7	33.2	2

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