

The Search for Adsorbed Gold Cyanide on Carbon Surfaces

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ABSTRACT

The mechanism of adsorption of gold from alkaline cyanide solutions by carbon has been the subject of much research over the past decade. In this paper, the nature of the gold cyanide adsorption phenomenon is investigated, particularly with respect to carbon substrates and associated surface adsorption sites.

INTRODUCTION

The use of activated carbon for the recovery of gold from alkaline cyanide solutions has been established for quite some time now, yet the actual mechanism of gold adsorption by carbon is not well understood. Comparatively little attention has been paid to the question of where on the surface of the carbon the gold is adsorbed, or in other words, the nature of the adsorption sites. The purpose of this paper is to investigate the nature of these adsorption sites, which should lead to a better understanding of the mechanism of gold adsorption by carbon.

BACKGROUND AND LITERATURE SURVEY

Current theories for gold adsorption by activated carbon favor adsorption of the gold cyanide complex without chemical change^(1,2). It has now been established that gold is not reduced to the metallic state on carbon during adsorption from alkaline cyanide solutions, as it is in the acidic chloride system.

Researchers at Mintek, in South Africa, maintain that adsorption of ion pairs is the predominant mechanism under the conditions prevalent in plant operations, that is, in high ionic strength alkaline solutions⁽¹⁾. At low ionic strength, they propose that ion exchange also becomes important for gold adsorption. However, these researchers do not address the question of where on the surface of the carbon the gold is adsorbed.

Jones and others⁽²⁾ have proposed that the gold cyanide complex is reversibly adsorbed by the hexagonal ring structure of the basal plane of graphite present to varying extents in activated carbon. Their analysis shows that the size of the gold cyanide complex is fairly well matched to that of the ring structure substrate of graphite. These researchers proposed that bonding occurs between the gold atom of the aurocyanide complex and the π electrons associated with the graphite ring structure. In essence, the mechanism proposed by Jones, et al.⁽²⁾ is adsorption without chemical change, which is in agreement to some extent with the results reported by researchers at Mintek. However, the Jones et al. XPS data do not support the formation of ion pairs. In this regard, it may be that the surface state of the adsorbed gold cyanide was altered by the drying and high vacuum required for the XPS procedure.

Miller and Sibrell⁽³⁾ investigated the adsorption of gold from alkaline cyanide solutions by a variety of different forms of elemental carbon, including activated carbon, carbon black, and graphite.

Their work has shown that the most important property of the carbon for gold adsorption is the graphitic structure associated with these materials. This was demonstrated vividly by the lack of gold adsorption by a sample of powdered diamond, which has a completely different crystallographic structure and bonding geometry than graphite. In the graphite structure, the carbon atoms are bound into infinite layers or planes of fused hexagonal rings. The layers are loosely held together by van der Waals forces. In the planar ring structure, the electrons are delocalized, which accounts for the high electrical conductivity of graphite in the basal plane. It is also possible that the gold cyanide complex interacts with the delocalized electrons, perhaps leading to formation of a chemical (π - donor) bond, which may explain the attachment of the gold cyanide complex to the graphite.

Adsorption density measurements made by these researchers⁽³⁾ showed that all of the graphitic carbon materials adsorbed roughly similar amounts of gold, when compared on a surface area basis. This is demonstrated in Figure 1, which shows adsorption on a surface area basis for activated carbon, carbon black and graphite. The adsorption density for all three types of carbon was on the order of 10^{-11} moles of $\text{Au}(\text{CN})_2^-$ per square centimeter of carbon surface over an equilibrium solution concentration range of 1 to 500 mg/L (ppm) gold. (Using BET surface areas for the activated carbon, carbon black, and graphite, of 1200 m^2/gm , 210 m^2/gm , and 3.7 m^2/gm , 10^{-11} moles per square cm gives 23,600 ppm, 4100 ppm, and 70 ppm Au adsorbed on the carbon, respectively.) This adsorption density corresponds to a coverage of about one tenth of a monolayer. The fact that adsorption densities were very similar for such dissimilar carbon materials suggests that the graphitic structure itself is the adsorption site. However, it could be that adsorption takes place at defects in the graphite structure, and the number of defects is directly related to the surface area of the material. Presumably, carbon atoms at defects would have unsatisfied valencies, or attached functional groups, that would react with the gold cyanide complex. To differentiate between these two possibilities, it will be necessary to learn more about the sites where the gold cyanide is adsorbed. This, in turn, will reveal more information about the mechanism of gold adsorption by carbon.

EXPERIMENTAL

MATERIALS

Several different types of carbon have been investigated in the search for adsorbed gold cyanide. Since activated carbon is used commercially for gold adsorption, this was a logical starting point. Three activated carbon samples were tested, each commonly used in commercial operations for the recovery of gold from cyanide pulps and leach solutions. These carbons were Norit RO 3515, an extruded carbon made from peat, and Calgon GRC-22 and NACAR (North American Carbon) G-204, both coconut-based carbons.

One disadvantage of activated carbon is its homogeneity, that is, all types of adsorption sites are distributed across the surface of the

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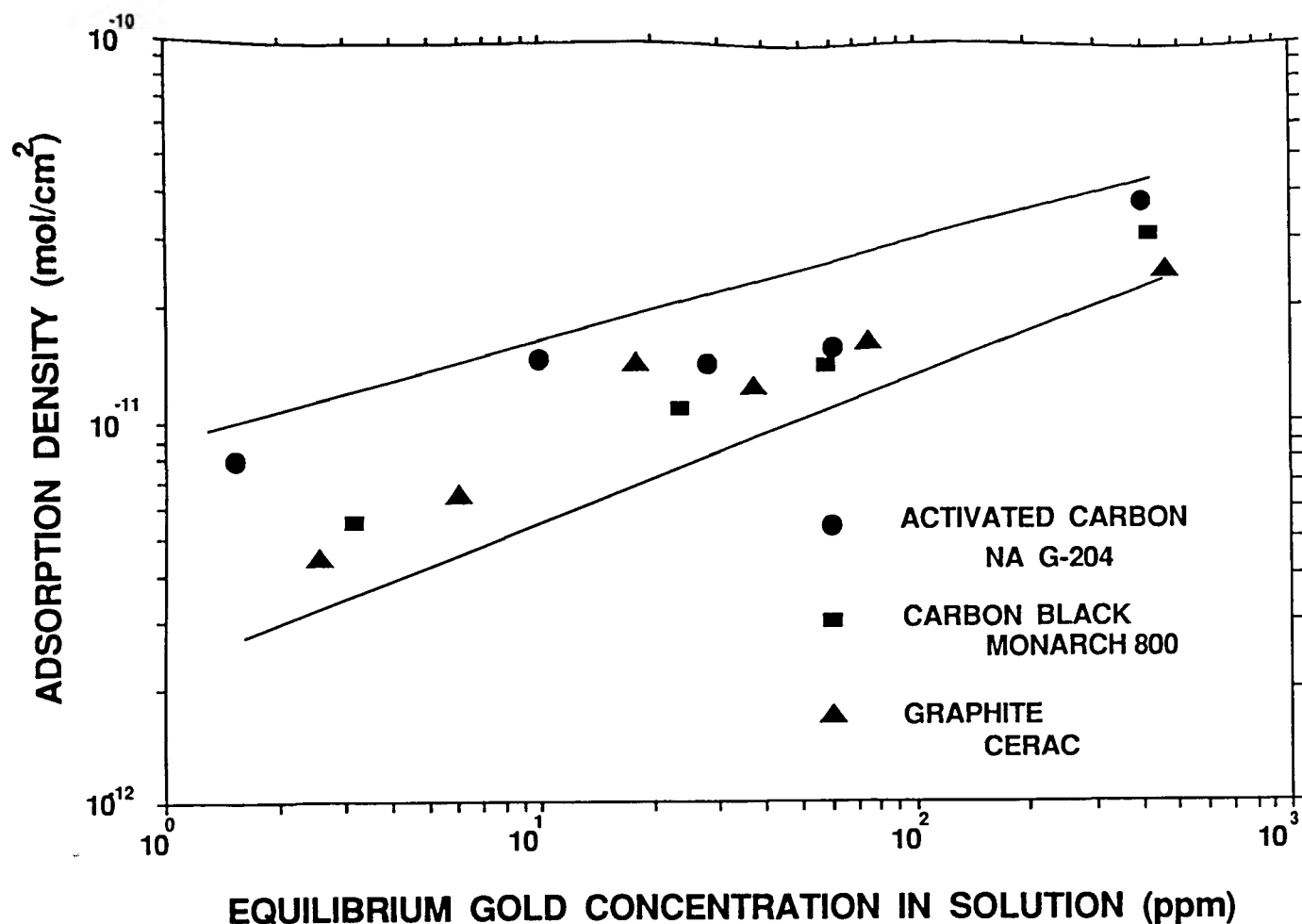


Fig. 1. Gold adsorption density as a function of equilibrium solution concentration for three different types of carbon.

carbon. It would be useful to be able to distinguish basal graphitic plane sites from edge, or defect sites, and in this regard, crystalline graphite is superior. Samples of lump graphite were obtained from Ward's Natural Science Establishment, in Rochester, New York. Although these samples were not perfect crystals, it was possible to determine the crystallographic orientation, and to polish these samples to give smooth basal plane sites.

An even more ideal graphite surface is presented by a synthetic material termed highly oriented pyrolytic graphite (HOPG). HOPG is manufactured in the United States by the Advanced Ceramics Division of Union Carbide. This material is produced by pyrolytic decomposition of methane onto a carbon substrate, followed by high temperature, high pressure annealing, which brings the layers of graphite into very close alignment, almost like a single crystal. However, it should be emphasized that this material is polycrystalline. The production and uses of HOPG are further detailed in a review by Moore⁽⁴⁾. HOPG presents an almost defect-free basal plane, and when turned 90°, an almost perfect array of basal plane edges.

Analytical grade reagents and deionized water were used for all laboratory work.

TECHNIQUES

Gold was adsorbed onto carbon samples by immersion of the sample in an alkaline gold cyanide solution, ranging in concentration from 100 to 5000 mg/L (ppm). In some cases, an inert electrolyte, such as calcium chloride, was added to the solution to

increase gold adsorption. With activated carbon, the gold loading could be calculated by analysis of the solution before and after contact, by DCP emission spectroscopy. For crystalline graphite and HOPG, the change in gold content of the solution was not detectable, due to the much lower capacity of these materials for gold adsorption.

FTIR Spectroscopy

Fourier Transform Infrared (FTIR) spectroscopy was used extensively in an earlier paper concerning the characterization of the carbonaceous matter in Carlin gold ores⁽⁵⁾. However, due to the low abundance of functional groups on the surface of the carbonaceous matter, the IR spectra were typically fairly featureless, with only two or three small peaks present. The problem gets progressively worse with activated carbon and graphite, not only due to the decreasing abundance of functional groups, but also because of electronic absorption of IR radiation at higher wavenumbers. This effect manifests itself as a sloping baseline in the spectrum.

Although it may not be feasible to determine the structure of the carbon itself by IR spectroscopy, it may be possible to track gold adsorption by these materials, via the $C\equiv N$ stretching peak at about 2140 cm^{-1} , due to the cyanide present in the gold cyanide complex. The traditional IR sampling method has been transmission spectroscopy, which is a bulk analysis of the sample. However, with the advent of Fourier Transform spectroscopy came many different possibilities for sampling, included those more adapted to observing the surface of the sample, by use of a number of reflective techni-

ques. These include internal reflection spectroscopy (IRS), diffuse reflectance, and external, or specular reflectance.

Infrared spectra of all samples were generated with a Digilab FTS 40 system. Each sample was scanned at least 256 times at a resolution of 4 cm^{-1} , and the scans coadded to obtain each spectrum.

Transmission Spectroscopy. In this method, 3 mg of pulverized sample was mixed with 300 mg of spectroscopic-grade potassium bromide. The sample and KBr were well mixed for five minutes in a Wig-L-Bug amalgamator, then 150 mg of the mixture was pressed in an evacuated die in a hydraulic press at 20,000 psi for five minutes. The resulting disk was placed in the spectrometer sample compartment, which was then purged for ten minutes to remove water vapor and carbon dioxide, after which the spectrum was taken.

Diffuse Reflectance. In this method, the IR radiation is reflected off a powder sample, which may or may not be diluted in a powdered IR-transparent matrix, such as potassium bromide. The carbon samples were run neat, or undiluted. The Collector diffuse reflectance accessory, manufactured by Spectra-Tech, Inc., was used for all diffuse reflectance measurements. This accessory fits in the spectrometer sample compartment, so that purging of the atmosphere around the accessory was done just as for transmission spectroscopy.

Internal Reflection. It would be advantageous to detect the CN band of gold cyanide in-situ as it adsorbs onto a carbonaceous material from solution. In theory, this should be possible using internal reflection spectroscopy. (This method is sometimes referred to as attenuated total reflectance spectroscopy.) In this technique, the IR radiation is focussed at the edge of an internal reflection element (IRE), and reflected internally a number of times before exiting. At each reflection, the radiation interacts with the material surrounding the crystal, but only to a depth of a few microns. This small sampling thickness allows spectra to be taken in aqueous solutions, which were very difficult to analyze in the past, due to the high absorbance of IR radiation by water.

The IRE was held in an attenuated total reflectance accessory, made by Spectra-Tech, Inc., which fitted into the sample compartment of the FTIR spectrometer. Solution was injected into the cell containing the IRE with a syringe from outside the sample compartment. Spectra were then taken at timed intervals after injection of the gold cyanide solution.

Specular Reflectance. In this sampling method, the IR radiation is reflected from a polished surface to the detector. This technique is appropriate for films or coatings on the polished surface. Theoretically, adsorbed species on the surface would also be picked up, provided the adsorption density was great enough. One disadvantage is that this technique is ex-situ, that is, the sample must be removed from solution. However, on the plus side, the orientation of the adsorbed species could be found using polarized light. The question of whether gold cyanide attaches to the basal plane or to edges of the basal planes could be addressed by preparing polished samples of graphite with different orientations, and measuring the density and orientation of adsorbed gold cyanide.

A variable angle specular reflectance accessory made by Spectra-Tech, Inc. was used for this work. The sample holder can accommodate variable angles of incidence of the IR beam. A high angle of incidence, a so-called grazing angle, gives a longer path-length through any films or coatings on the polished surface, but the beam is spread out, and due to the small size of the samples, much of the beam did not reflect from the sample. Therefore, a compromise angle of incidence of 40° was selected.

Electrochemistry

Another method which could prove useful in the detection of adsorbed gold cyanide on carbon surfaces is electrochemical analysis. This procedure involves adsorption of gold cyanide from solution onto a carbon electrode, followed by a cathodic scan to reduce the adsorbed gold cyanide to metallic gold. The current associated with the reduction of the gold cyanide would then be proportional to the amount of gold adsorbed. For the expected adsorption density of gold cyanide, this current will be small, on the order of microamperes, but modern potentiostats are capable of detecting this amount of current. The electrode would be rinsed and transferred to another conductive solution following gold adsorption to eliminate the presence of gold cyanide in the solution phase. Alternatively, it may not be necessary to transfer the electrode to another solution if a sufficiently high scan rate was used. In this case, the current due to adsorbed species would be discharged immediately, whereas there would be a time delay before diffusing ions could be reduced at the electrode. Initially, a glassy carbon rotating disk electrode was used for gold adsorption experiments. This material has a random orientation of basal planes and edges.

Two electrochemical techniques have been frequently used for the detection of adsorbed species. These are cyclic voltammetry⁽⁶⁾, and chronocoulometry⁽⁷⁾. In cyclic voltammetry, the input voltage is varied at a constant rate, and the associated current recorded as a function of the voltage on an X-Y recorder. In chronocoulometry, the voltage is stepped to a selected value, and the charge, or the integral of the current-time curve, is recorded as a function of elapsed time after the voltage step.

The electrochemical equipment used in this research was made by the Princeton Applied Research Corporation (PARC), and included a Model 173 potentiostat, Model 376 current converter, Model 175 universal programmer, and a Model 379 digital coulometer. Both the rotating disk electrode and the rotator were made by the Pine Instrument Company. Resultant waveforms were recorded on a Bausch and Lomb X-Y recorder. For chronocoulometric measurements, a Hewlett-Packard Model 130C oscilloscope with a Polaroid camera was used to record the current and charge response. Later, a PARC Model 276 computer interface was added to the potentiostat to enable computer storage and manipulation of the output waveform. All electrochemical work was done in a three-neck flask, with a saturated calomel reference electrode, and a platinum counter electrode, in addition to the carbon working electrode. The cell and solution were purged with nitrogen to remove dissolved oxygen from solution before the test was begun.

Radiochemistry

The sensitivity of radiochemical methods could be a great advantage in the problem of detecting adsorbed gold cyanide on the surface of graphitic carbon. If the adsorbed gold cyanide species were labeled with a radioactive isotope, adsorption densities could be measured directly with a radiation counter. Accordingly, it was decided to prepare a solution containing radiolabeled gold cyanide by the dissolution of gold in a tagged potassium cyanide solution. The potassium cyanide was labeled with the carbon-14 isotope, which emits beta radiation with a maximum energy of 0.155 MeV, and has a half life of 5730 years⁽⁸⁾. The tagged potassium cyanide was obtained from the Sigma Corporation with an activity of about 50 Curies (Ci) per mole. (One Ci of a radioactive material undergoes 2.22×10^{12} disintegrations per minute (dpm)).

The radiolabeled potassium cyanide was dissolved in a small volume of water that had previously been adjusted to pH 10 with

sodium hydroxide. Metallic gold (Alfa Products, 99.99 per cent) was added to the solution at two times the stoichiometric amount to ensure that all of the cyanide was bound as gold cyanide. Aliquots of this solution were evaporated to dryness in counting planchets for preparation of a calibration curve for the counter. Samples of HOPG were sealed into epoxy disks with either basal plane or edge orientations. Basal plane HOPG surfaces were prepared by removing the top layer with transparent tape, and edge surfaces were prepared by polishing with alumina slurries on a rotating polishing wheel. The HOPG samples were then exposed to the radiolabeled gold cyanide solution overnight, rinsed thoroughly, then placed in a planchet for counting. The radiation was counted with a Canberra gas flow proportional detector, with an anticoincidence circuit to reduce the background count rate.

RESULTS

FTIR SPECTROSCOPY

Transmission Spectroscopy

The first sample examined by transmission spectroscopy was powdered activated carbon that had been contacted with gold cyanide solution to give an adsorbed gold loading of about 9 per cent as indicated from solution analysis. As a comparison, powdered potassium gold cyanide salt was mixed with powdered activated carbon to give two samples of 6 per cent and 30 per cent gold by weight. The FTIR transmission spectra for all three of these samples is shown in Figure 2.

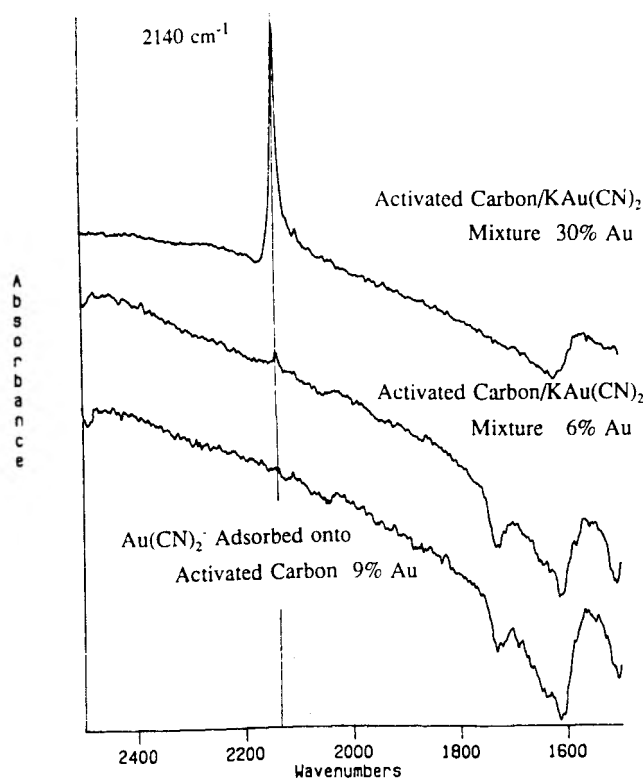


Fig. 2. FTIR transmission spectra of activated carbon showing a comparison of mixtures of potassium gold cyanide salt with adsorbed gold cyanide.

As can be seen, the cyanide peak at 2140 cm^{-1} was present in both samples prepared as mixtures. However, for the case of the adsorbed gold cyanide, no cyanide peak was detectable, even though the concentration of gold was higher than that of the mixture sample containing 6% Au. This may indicate that the adsorbed gold is hidden in the pore structure of the activated carbon, inaccessible to the IR radiation.

To ensure that this was not an isolated effect, the experiment was repeated, this time using three different activated carbon samples, including North American G-204, Calgon GRC-22, and Norit RO 3515. Also, the gold loading was increased by using higher gold concentrations in solution (5 grams per liter or gpl), and by the addition of 0.5 M calcium chloride, which is known to enhance gold adsorption. The gold loading on these samples was calculated to be over 20% Au by weight, from solution analysis. The resulting transmission spectra are shown in Figure 3.

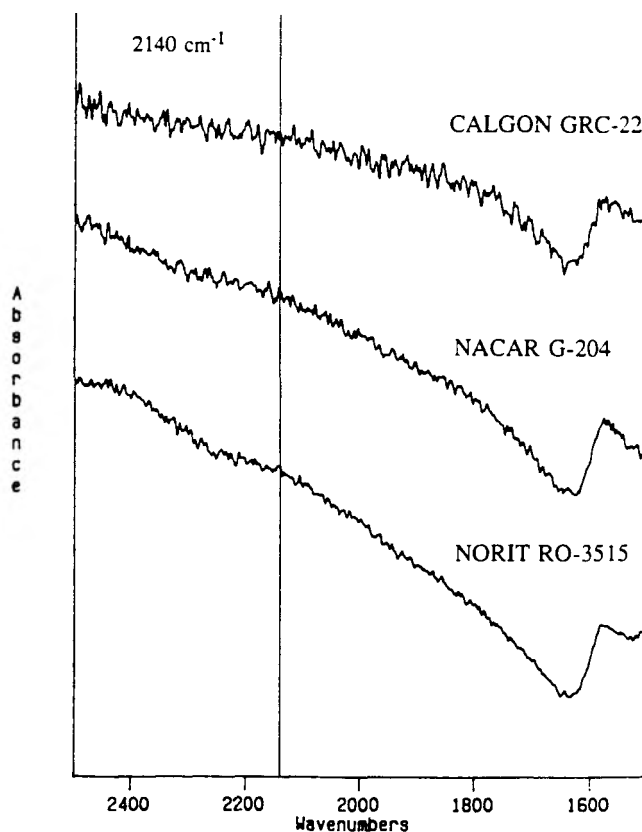


Fig. 3. FTIR transmission spectra of activated carbon samples after adsorption of gold cyanide. Gold concentration is at least 20% by weight for all three samples.

As can be seen, no cyanide peaks were detected, even at these high gold loadings. In contrast, the peaks at about 1580 cm^{-1} are due to the graphitic ring structure, and are clearly shown in all three of the samples. Even when the bands due to the activated carbon itself are well defined, the adsorbed gold cyanide cannot be detected.

Diffuse Reflectance

The results from transmission spectroscopy suggest that the adsorbed gold cyanide may be in the pore structure of the activated carbon, where the IR radiation cannot penetrate. Samples with lower porosity should have more gold cyanide on the surface. Therefore, samples of carbon black and graphite were contacted with alkaline gold cyanide solutions, filtered, washed, dried, and sampled neat by diffuse reflectance. For the carbon black, the BET

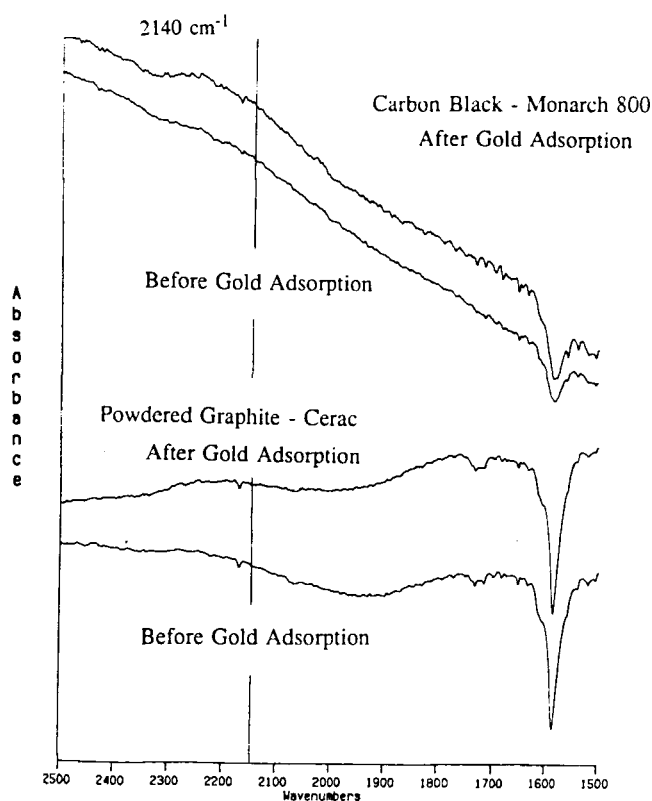


Fig. 4. FTIR diffuse reflectance spectra of carbon black and graphite samples after adsorption of gold cyanide.

surface area was 210 m²/gm and the gold loading was 1.5% Au by weight. For the graphite sample, the surface area was 3.7 m²/gm, and the gold loading, 0.02% by weight. The resulting spectra are shown in Figure 4.

Again, as was the case with transmission spectroscopy, the cyanide peak was not detected for either sample, even though the ring stretching band at 1580 cm⁻¹ was clearly shown for both samples.

Internal Reflectance

For this work, an inert internal reflectance element (IRE) made of calcium fluoride (transparent to radiation from 50,000 to 1140 cm⁻¹) was coated with a thin layer of finely divided activated carbon. The application of a thin, adherent layer of carbon to the IRE required some experimentation. It was found that good coatings could be applied by repeatedly dipping the IRE into a slurry of activated carbon in acetone, and allowing the acetone to evaporate between dips. In this case, the IRE was dipped into the slurry about 50 times, which resulted in a coating which was still translucent to light. From the increase in weight of the IRE after coating, the thickness of the coating was calculated to be on the order of 100 μm. The coated IRE was then placed in the internal reflectance accessory, and exposed to pure water (to be used as a reference for subtraction), and finally, exposed to a solution containing gold cyanide.

Although the coated IRE was exposed to the solution for over 72 hours, no CN peaks were detected. Since the background water spectrum showed low absorbance, it is possible that the carbon coating was too thick, and reflected the majority of the IR radiation before it ever reached the activated carbon/solution interface.

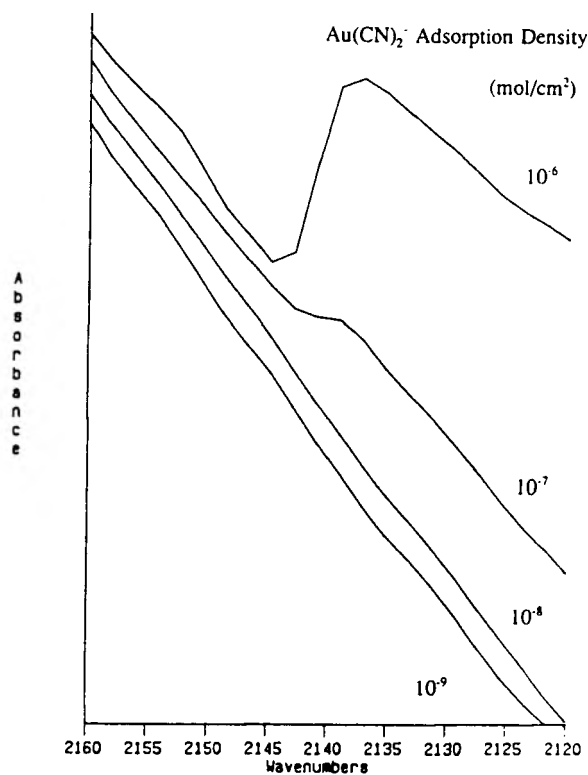


Fig. 5. FTIR specular reflectance spectra of potassium gold cyanide salt evaporated onto polished surfaces of natural graphite

Specular Reflectance

Several different carbon types were polished for initial testing by specular reflectance. These carbons included natural crystalline graphite and highly oriented pyrolytic graphite (HOPG). The samples were then contacted with an alkaline solution containing 5000 ppm Au (no background electrolyte) for several days, rinsed, dried, and specular reflectance spectra taken. Initially, it appeared that adsorbed gold cyanide had been detected on one of the samples, as a strong peak at 2140 cm⁻¹ was observed for that sample. However, subsequent investigation showed that crystalline salts were present on the surface of this sample, and it was probable that the gold cyanide solution had penetrated the structure of the sample, then come to the surface and dried when the sample was removed from solution. The resulting salt crystals gave a cyanide peak in the IR spectrum, but this was not due to adsorbed species.

At this point, it was decided to determine just what the detection limit for gold cyanide on a carbon surface would be. This was done by evaporation of aliquots of gold cyanide solutions of known strength onto a polished graphite surface, and examination of the resulting specular reflectance spectra to see what the detection limit for the cyanide species was. Although the gold cyanide was present as a salt, rather than as an adsorbed species, still it would be possible to estimate from this data whether monolayer coverage of adsorbed gold cyanide would be detectable. The results are shown in Figure 5.

The smallest concentration of gold cyanide detectable on the surface of the graphite was on the order of 10⁻⁷ moles per cm². As discussed earlier, it was found that even at fairly high loadings,

carbon samples have about 10^{-11} moles of gold cyanide per square cm of carbon surface area. From this analysis, it appears that IR spectroscopy is not nearly sensitive enough to detect adsorbed gold cyanide under these conditions.

ELECTROCHEMISTRY

Initially, cyclic voltammetry was investigated as a possible technique for the detection of adsorbed gold cyanide on the surface of a glassy carbon rotating disk electrode. In this regard, the first step was to study the reduction of gold cyanide from solution, for familiarization with this system. The peak for gold reduction on the carbon electrode was found to be about -1.6 V vs the saturated calomel electrode (SCE). The equilibrium reduction potential for this reaction is -0.81 V vs SCE, as calculated from the standard potential of -0.87 V vs SCE, with adjustments for the concentrations of the cyanide and aurocyanide species in solution. (The SCE has a potential of +0.241 V with respect to the normal hydrogen electrode.) Evidently, there is a large overpotential associated with reduction of the gold on the carbon surface. Unfortunately, the reduction potential is uncomfortably close to the reduction potential for the evolution of hydrogen on the electrode, even at alkaline pH. Further work showed that the current associated with the reduction of the adsorbed gold cyanide was so small (about one microampere, or less) that the charging current associated with the capacitive double layer at the electrode began to interfere with the measurement. The charging current is proportional to the scan rate, but slow scan rates are not feasible because slow discharge of electrons due to the reduction of gold cyanide would give a much lower current, as current is charge per unit time. The effect of the charging current is shown in Figure 6, which shows a cyclic voltammogram of the glassy carbon electrode in an inert electrolyte.

It is clear from this figure that the detection of one microampere of current due to adsorbed gold species will not be possible under these conditions, due to the large background charging current.

An alternative electrochemical technique for the detection of adsorbed species on an electrode is chronocoulometry. With this technique, adsorbed species can be distinguished from diffusing species by the time dependencies of the reaction at the electrode. This is because diffusing species migrate to the electrode at a rate proportional to the square root of time, while species adsorbed on the electrode are reduced almost immediately. Unfortunately, the capacitance of the double layer comes into effect here also, and is manifested in precisely the same way as the reduction of the adsorbed species, that is, as an almost instantaneous flow of charge when the potential is stepped to the reduction value. Due to this complication, it was not possible to measure the quantity of adsorbed gold cyanide on the glassy carbon electrode.

The double layer capacitance effects that interfered with the electrochemical detection of adsorbed gold cyanide can be reduced by using an electrode with a lower capacitance. Highly oriented pyrolytic graphite (HOPG) basal plane electrodes are known for their low capacitance of about $3 \mu\text{F}/\text{cm}^2$, compared to about $13 \mu\text{F}/\text{cm}^2$ for glassy carbon⁽⁹⁾. Therefore, further work in this area should be done using HOPG electrodes.

RADIOCHEMISTRY

The expected activity of the gold cyanide adsorbed on the HOPG samples can be calculated as follows. As discussed earlier, the expected gold cyanide adsorption densities are on the order of 10^{-11} moles per square cm of graphite surface. HOPG samples were prepared with about one square cm of exposed area. Radiolabeled potassium cyanide was obtained from the Sigma Corporation with an activity of about 50 Curies (Ci) per mole. Since one Ci cor-

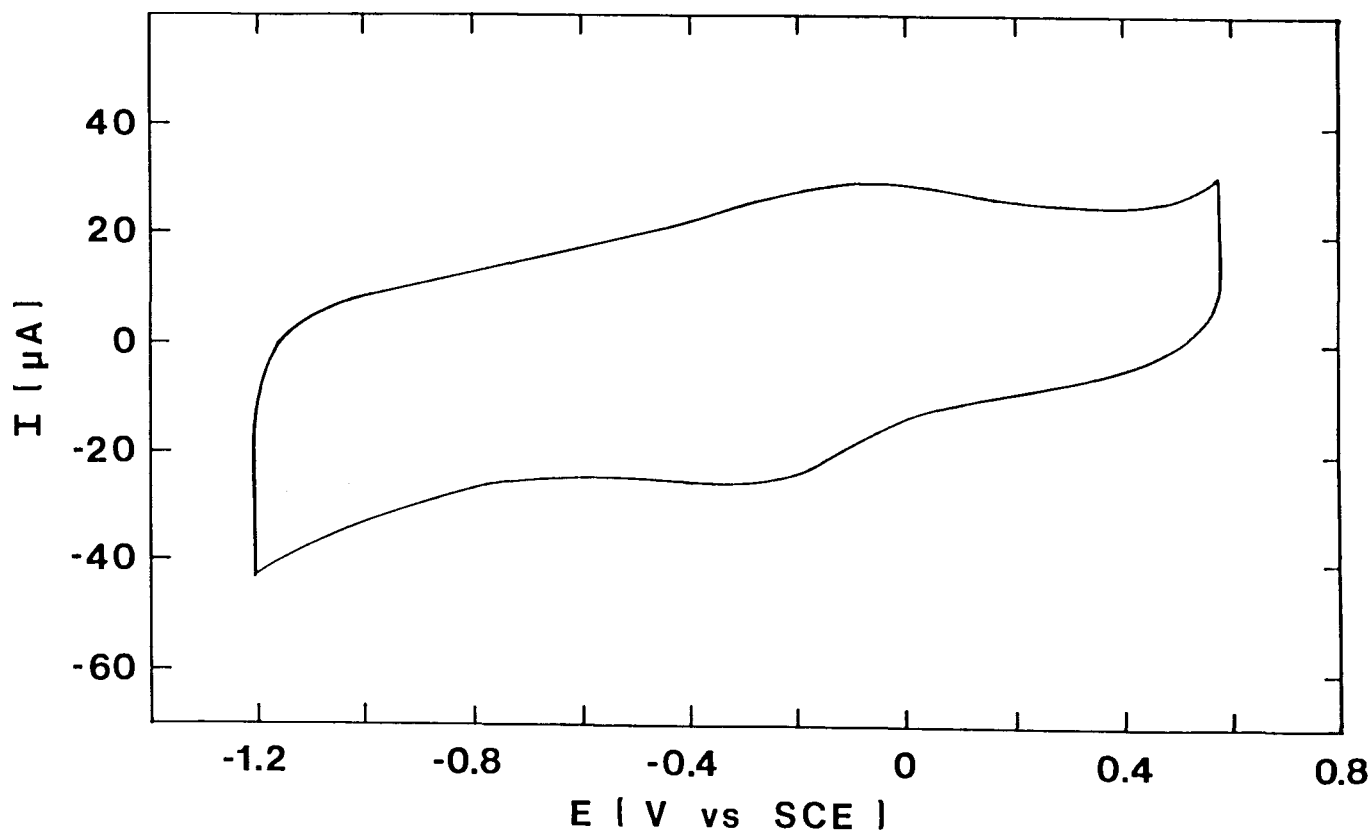


Fig. 6. Cyclic voltammogram of glassy carbon rotating disk electrode in 1 M KNO_3 oxygen-free electrolyte solution. Scan rate = 100 mV/s, rotation speed = 190 RPM, electrode area = 0.46 cm^2 .

responds to 2.22×10^{12} disintegrations per minute, a loading of 10^{-11} moles of gold cyanide would be equivalent to 10^{-9} Ci, or an activity of 2220 disintegrations per minute. The radiation was counted with a Canberra gas flow proportional detector, with an anticoincidence circuit to reduce the background count rate to about 2 counts per minute. Due to adsorption of the radiation by air and the counter window, counting efficiencies were on the order of 1 per cent, which gives a count rate of about 22 counts per minute. This is significantly greater than the background count rate (2 counts per minute) of the counter, and thus should be measurable.

A calibration curve was prepared by evaporating aliquots of radiolabeled solution into counting planchets. The calibration curve is shown in Figure 7.

The upper curve in Figure 7 shows the disintegration rate of the radiolabeled gold cyanide. The lower series of points shows the actual count rate detected by the counter. The actual count rate is about 1 per cent of the disintegration rate, due to efficiency of the counter and other factors. As discussed above, the HOPG samples should have on the order of 10^{-9} Ci of C-14 on the surface, which from the calibration curve, will correspond to about 20 counts per minute. Since the background counting rate on this counter is about two counts per minute, adsorbed species at the anticipated level should be detected. Unfortunately, at the time of writing of this publication, this work was not complete, but experiments are proceeding in our laboratory along these lines. This method has the most promise for resolution of the nature of the adsorption sites for gold cyanide on carbon surfaces.

SUMMARY

The search for adsorbed gold cyanide species on carbon surfaces was undertaken using IR spectroscopic techniques, electrochemical techniques, and radiolabeling methods. Of these methods,

radiolabeling appears to offer the best chance for the detection of adsorbed gold cyanide, and work is proceeding in our laboratory along these lines. It appears that IR spectroscopy, under these conditions, does not have the sensitivity necessary to detect adsorbed gold cyanide on carbon. Electrochemical techniques showed promise, but due to double layer capacitance effects, were unsuccessful in detecting gold cyanide. Perhaps with the lower capacitance HOPG basal plane electrode, detection will be possible.

Other analytical techniques may also be of use. For example, X-ray photoelectron spectroscopy (XPS) is known to be sensitive for the presence of metals on a surface, and has in fact been used for analysis of gold loaded activated carbons. This technique would be one possibility for an independent verification of the results from the radiotracer work. It would be even more helpful if an analytical technique could be found that would give the distribution of gold cyanide species on the carbon surface, which could then be correlated to the topography of the surface.

The determination of the nature of the gold cyanide adsorption sites on carbon surfaces will reveal much information about the adsorption mechanism itself. If gold adsorption densities are found to be greater on graphitic basal plane sites, it would indicate that gold adsorption on carbon is generally non-specific, that is, all such adsorption sites are equivalent. On the other hand, if adsorption densities are found to be greater at edge sites, it would suggest that adsorption is selective, or that certain sites are favored over others, due to different geometries or bonding characteristics.

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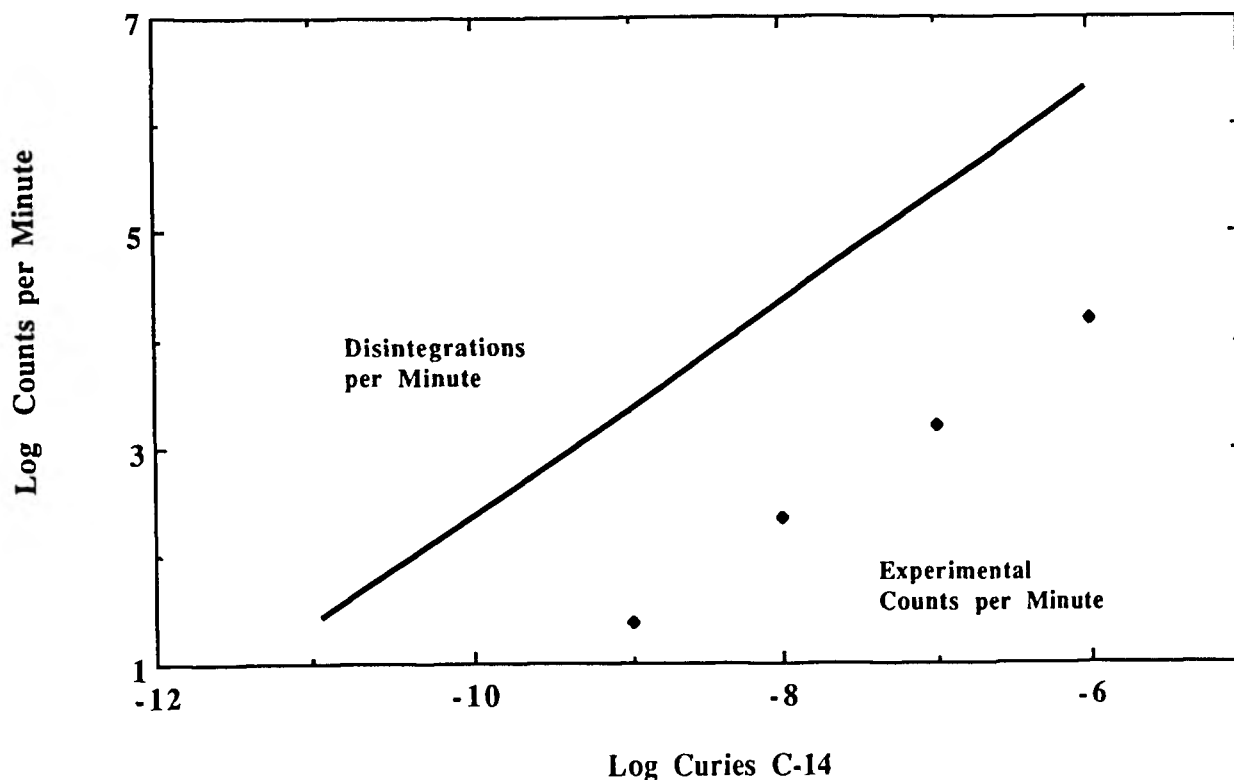


Fig. 7. Calibration of gas flow proportional radiation counter for analysis of carbon-14 labeled gold cyanide samples.

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