



Ion-Exclusion Chromatography for Analyzing Organics in Water

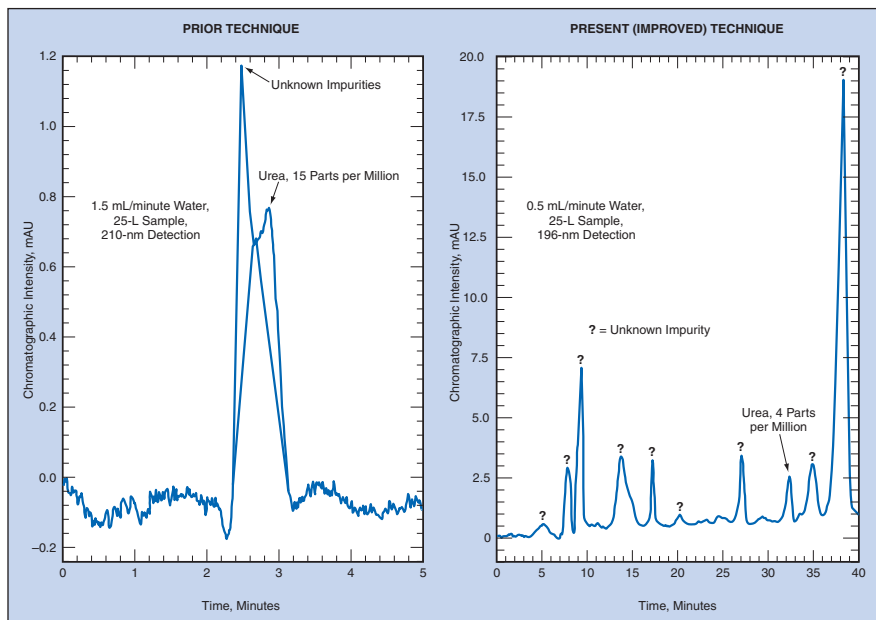
Resolution of nonvolatile organic compounds is increased significantly.

Lyndon B. Johnson Space Center, Houston, Texas

A liquid-chromatography technique has been developed for use in the quantitative analysis of urea (and of other nonvolatile organic compounds typically found with urea) dissolved in water. The technique involves the use of a column that contains an ion-exclusion resin; heretofore, this column has been sold for use in analyzing monosaccharides and food softeners, but not for analyzing water supplies.

The prior technique commonly used to analyze water for urea content has been one of high-performance liquid chromatography (HPLC), with reliance on hydrophobic interactions between analytes in a water sample and long-chain alkyl groups bonded to an HPLC column. The prior technique has proven inadequate because of a strong tendency toward co-elution of urea with other compounds. Co-elution often causes the urea and other compounds to be crowded into a narrow region of the chromatogram (see left part of figure), thereby giving rise to low chromatographic resolution and misidentification of compounds. It is possible to quantitate urea or another analyte via ultraviolet- and visible-light absorbance measurements, but in order to perform such measurements, it is necessary to dilute the sample, causing a significant loss of sensitivity.

The ion-exclusion resin used in the improved technique is sulfonated polystyrene in the calcium form. Whereas the alkyl-chain column used in the prior technique separates compounds on the basis of polarity only, the ion-exclusion-resin column used in the improved technique separates compounds on the basis of both molecular size and electric



These Chromatograms were made from samples of space-shuttle humidity condensate, which contained dissolved urea and other impurities.

charge. As a result, the degree of separation is increased: instead of being crowded together into a single chromatographic peak only about 1 to 2 minutes wide as in the prior technique, the chromatographic peaks of different compounds are now separated from each other and spread out over a range about 33 minutes wide (see right part of figure), and the urea peak can readily be distinguished from the other peaks.

Although the analysis takes more time in the improved technique, this disadvantage is offset by two important advantages:

- Sensitivity is increased. The minimum concentration of urea that can be

measured is reduced (to between 1/5 and 1/3 of that of the prior technique) because it is not necessary to dilute the sample.

- The separation of peaks facilitates the identification and quantitation of the various compounds. The resolution of the compounds other than urea makes it possible to identify those compounds by use of mass spectrometry.

This work was done by Richard Sauer of Johnson Space Center and Jeffrey A. Rutz and John R. Schultz of Wyle Laboratories. For further information, contact the Johnson Commercial Technology Office at (281) 483-3809. MSC-23000

Selective Plasma Deposition of Fluorocarbon Films on SAMs

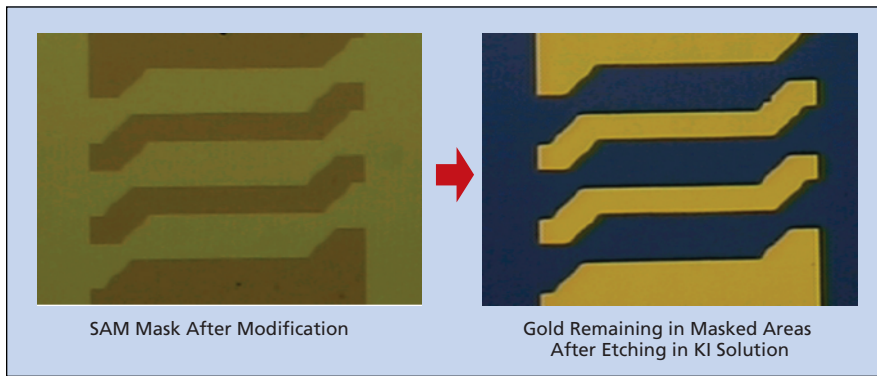
The fluorocarbon films are useful as etch masks and perhaps as dielectric layers.

Goddard Space Flight Center, Greenbelt, Maryland

A dry plasma process has been demonstrated to be useful for the selective modification of self-assembled monolayers (SAMs) of alkanethiolates.

These SAMs are used, during the fabrication of semiconductor electronic devices, as etch masks on gold layers that are destined to be patterned and incor-

porated into the devices. The selective modification involves the formation of fluorocarbon films that render the SAMs more effective in protecting the masked



A **Gold Layer Was Patterned** by stamping it with a hexadecanethiol solution. The alkanethiolate SAM pattern thus formed was modified in a CF_4/H_2 plasma, forming an etch mask. Then gold was etched away from the areas not masked.

areas of the gold against etching by a potassium iodide (KI) solution. This modification can be utilized, not only in the fabrication of single electronic devices but also in the fabrication of integrated circuits, microelectromechanical systems, and circuit boards.

In the steps that precede the dry plasma process, a silicon mold in the de-

sired pattern is fabricated by standard photolithographic techniques. A stamp is then made by casting polydimethylsiloxane (commonly known as silicone rubber) in the mold. The stamp is coated with an alkanethiol solution, then the stamp is pressed on the gold layer of a device to be fabricated in order to deposit the alkanethiol to form

an alkanethiolate SAM in the desired pattern (see figure). Next, the workpiece is exposed to a radio-frequency plasma generated from a mixture of CF_4 and H_2 gases. After this plasma treatment, the SAM is found to be modified, while the exposed areas of gold remain unchanged.

This dry plasma process offers the potential for forming masks superior to those formed in a prior wet etching process. Among the advantages over the wet etching process are greater selectivity, fewer pin holes in the masks, and less nonuniformity of the masks. The fluorocarbon films formed in this way may also be useful as intermediate layers for subsequent fabrication steps and as dielectric layers to be incorporated into finished products.

This work was done by Mark M. Crain III, Kevin M. Walsh, and Robert W. Cohn of the University of Louisville for Goddard Space Flight Center. Further information is contained in a TSP (see page 1). GSC-14440

Water-Based Pressure-Sensitive Paints

Toxicity, and thus costs of ventilation and cleanup, is substantially reduced.

Langley Research Center, Hampton, Virginia

Water-based pressure-sensitive paints (PSPs) have been invented as alternatives to conventional organic-solvent-based pressure-sensitive paints, which are used primarily for indicating distributions of air pressure on wind-tunnel models. Typically, PSPs are sprayed onto aerodynamic models after they have been mounted in wind tunnels. When conventional organic-solvent-based PSPs are used, this practice creates a problem of removing toxic fumes from inside the wind tunnels. The use of water-based PSPs eliminates this problem. The water-based PSPs offer high performance as pressure indicators, plus all the advantages of common water-based paints (low toxicity, low concentrations of volatile organic compounds, and easy cleanup by use of water).

A typical PSP (whether conventional or of the present innovative type) contains the following:

- A luminescent compound, the luminescence of which is quenched by oxygen;
- An oxygen-permeable polymeric binder;
- Pigment materials to hide the painted surface and to increase reflectance of

the light emitted from the luminescent compound; and

- Solvents.

The paint is applied to a surface of interest on a model and allowed to dry. During a subsequent wind-tunnel test, under the appropriate illumination, the intensity of luminescence emitted by the

PSP is inversely proportional to the concentration of oxygen, hence, to the air pressure at the painted surface.

A water-based PSP contains all the basic ingredients of a conventional PSP (see table), except that the organic-solvent content (if any) is much smaller and the basic ingredients are incorpo-

Ingredient	Proportion in Weight Percent
water emulsion of poly-trifluoroethylmethacrylate-co-isobutylmethacrylate (1:1)	42 to 68.4
propylene glycol	2.28
a glycol ether	10.26
TiO ₂ (pigment)	11.35
N-methyl pyrrolidinone	6.00
Byk 346 (or equivalent solution of polyether-modified dimethylpolysiloxane)	0.68
Lubrizol 2062 (or equivalent) aliphatic/aromatic phosphate ester for solvent-borne and water-borne coatings	1.03
platinum tetra(pentafluorophenyl)porphyrin (a luminophore)	0.1

A **Typical Water-Based PSP Formulation** is shown here.