

**The****SPEX**

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**Speaker****S P E X HIGH PURITY INORGANICS**

(their preparation and characterization)

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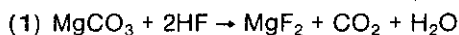
**T**HE meaning that the term "high purity" conjures up to each of our readers is likely to be as different as their individual specializations. To an emission spectrographer, potassium nitrate is high purity when it is free of metallic contamination, instrumentation conveniently ignoring all else, including organics and anions. To an electrical engineer, oxygen-free is the keyword for the desirable high-conductivity copper. To the optics designer, spectrographic grade 99.999% MgF<sub>2</sub> with less than 10 ppm cationic impurities is unsatisfactory if it contains an appreciable amount of MgO, a possible product of the drying process. To the solid state physicist, defect structure and stoichiometry are the essential parameters, while still others are concerned with isomers, isotopes or mixtures.

In addition to the purity, the method of preparation may dictate the applicability of a compound. Silver chloride, normally a soft, ungrindable material, can be specially crystallized into a friable spectrographic buffer of identical purity. Palladium oxide, made from the metal and oxygen at 700C, can wind up inactive whereas, prepared by decomposition of the nitrate at 500C, it becomes a good hydrogenation catalyst. From whomever the call comes for a high-purity material, on us falls the total task of filling the bill with compounds suitably prepared, characterized and packaged.

**PREPARATION**

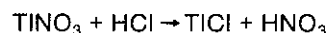
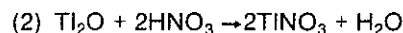
In practice, the synthesizing method is determined by the purity of the starting material and the planned application. Zone or electrolytic refining commonly yield 4 to 6-9s pure metals and oxides can then be prepared by direct reaction with oxygen or by pyrolysis of a salt in the form of a nitrate, carbonate, or oxalate. As long as the reagents contain no significant metallic impurities and are reacted in an inert vessel, the oxide product will test as pure spectrographically as the metal.

Acid/base reactions are good for compounds like magnesium fluoride for which the products are carbon dioxide or water:



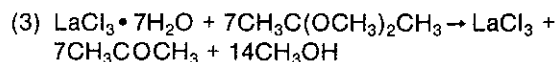
Again the product will be as spectrographically pure as the starting material and the reacting acid. To prevent partial conversion to an oxide, however, the compound must be dried in a stream of HF gas rather than air.

Precipitated from an aqueous solution, some compounds such as thallium chloride will be purer than the starting material:



Improvement results from the fact that the insoluble TiCl can be separated by filtration and many of the trace elements in the Ti<sub>2</sub>O such as Zn, Cu, Mg, and Fe will remain in the filtrate since their respective chlorides are water soluble. Of course, elements such as Pb or Ag which form insoluble chlorides will remain in the TiCl.

For anhydrous compounds simple heating at low temperatures will sometimes remove water of hydration. At other times organic dehydrating agents such as 2,2-dimethoxypropane [1] will be required to prevent hydrolysis reactions which form mixed compounds:

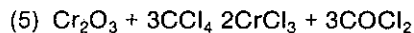


The resulting weakly coordinated methanol and acetone is easily removed by pumping under vacuum; no impurities are introduced.

In the solid state, high-purity materials can be synthesized by a direct combination of elements:



or by replacement reactions with organic or inorganic gases:



In all of these reactions, the purity of the final product is highly dependent upon a large number of factors besides the purity of the starting material. One is the choice of reaction vessel which is of utmost importance in maintaining a product free of contaminants. Table 1 lists some common container materials along with their major and minor components.

**Table 1**

Container Material	Major Components	Trace Components
Borosilicate Glass	Si, Al, Na, K, B, Ca, Mg	Numerous
Vycor	Si, Al, B	Numerous
Vitreous Silica	Si	Al, Ca, Mg, Ti
Graphite	C	V, Cu, Si, Mg
Platinum	Pt	Noble metals
Organic Polymers	C, H	Na, Al, Zn, P, Ca
Teflon	C, F	Na

In general, borosilicate glass containers are to be avoided since many elements can be leached from them particularly in acid or alkaline solutions. Vycor and vitreous silica offer some improvement, the latter even for high-temperature ignitions during which only small amounts of silica will be introduced. Graphite is a good choice where reduction is not a problem; however, most heavy-metal compounds will be reduced to the metal at elevated temperatures in a graphite crucible. At high temperatures platinum releases the fewest impurities but it does react with non-metals, molten metals and certain oxidizing agents. Refractories like  $\text{Al}_2\text{O}_3$  are ideally ignited in crucibles of their own substance. Polyethylene, polypropylene and other organic polymers are fine at room temperature although they may harbor large varieties of trace metal compounds introduced as mold releases, oxidation inhibitors, etc. Precleaning new containers with an acid solution is recommended. Teflon containers have few trace impurities and offer the advantage of better thermal stability. Steer clear of colored plastics, however; white comes from  $\text{TiO}_2$  and brown from  $\text{Fe}_2\text{O}_3$  fillers.

Sample treatment such as filtration is another potential source of contamination. Porous polyethylene pads in a Buchner funnel are fine for coarse or crystalline precipitates but Teflon fiber filter pads are needed for finer precipitates. Not recommended, of course, are sintered glass or conventional filter paper and funnels. To minimize contamination from atmospheric dust which circulates a considerable amount of iron, silica and other materials, available protective equipment ranges from covered crystallizing dishes (Thiers assembly) and glove boxes to clean rooms with lamellar flow hoods [2] The proper equipment selected, consideration must be given to the choice of reagents.

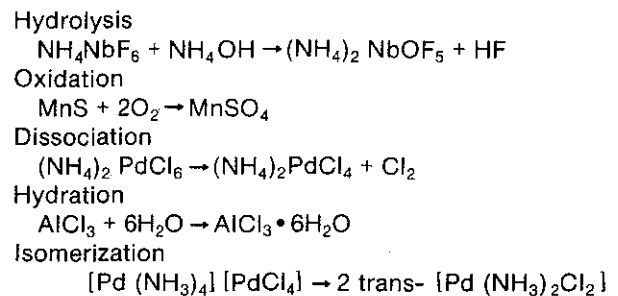
Distilled-deionized water is usually sufficiently pure but membrane filters will reduce the amount of suspended material even further. Among today's reagents HCl,  $\text{HNO}_3$  and  $\text{H}_2\text{SO}_4$  ordinarily have less than 1 ppm of metallic impurities. Exceptions include silicon, found at up to 10 ppm in hydrofluoric acid and anionic impurities of up to 500 ppm HCl in reagent grade HBr. Special acids, commonly marketed for trace mercury analysis, however, are usually purer by at least an order of magnitude.

When salts are added to precipitate a compound from solution the best cation is the ammonium ion since its residual ions will not interfere with a spectrographic analysis and its salts can easily be removed if necessary by volatilization at elevated temperatures. Whenever possible it is preferable to precipitate a compound from solution, leaving impurities in the filtrate, rather than evaporate to dryness, concentrating them in the product. However, coprecipitation of impurities can also affect the final compound considerably. In precipitating  $\text{BaSO}_4$ , for example, cationic impurities in the solution, such as Ca, Sr, or Pb which form slightly soluble sulfates, will be removed from solution and concentrated in the  $\text{BaSO}_4$ . Homogeneous precipitation from solution or digestion of the precipitate will sometimes, but not always, mitigate this problem. A related effect is that of post-precipitation, in which, after an induction period, impurities concentrate on the surface of a precipitate even though they are soluble

in the reaction media [3]. Ion entrapment can also occur, where soluble salts become trapped in the lattice of a precipitated compound. A good example is the entrapment of 1%  $\text{KNO}_3$  in a precipitate of  $\text{CaCrO}_4$  formed by the addition of  $\text{K}_2\text{CrO}_4$  to  $\text{Ca}(\text{NO}_3)_2$ . The  $\text{KNO}_3$  cannot be removed, even by digestion of the precipitate in hot water for several days although it is very soluble in water.

Other major sources of impurities are side reactions which can often yield mixtures of products [4]. As summarized in Table 2, in all cases the mixture of products would appear identical spectrographically to the single component but the physical and chemical properties of the side products would differ considerably from those of the major component.

Table 2



Even where a single compound results, it may not be stoichiometric. Many metal compounds, such as the oxides of Mn, Ni, Co, Tb, and Pr, have variable stoichiometries depending upon the temperature and oxygen pressure at which they were prepared. The transition from  $\text{Pr}_2\text{O}_3$  to  $\text{PrO}_2$  can proceed with little change in crystal structure since  $\text{PrO}_2$  has an eight-coordinate  $\text{CaF}_2$  structure, while  $\text{Pr}_2\text{O}_3$  has a similar structure with approximately 25% of the oxygen sites vacant [5].

Stoichiometry can also vary with hydration, and adsorption of moisture or carbon dioxide from the air. A recent off-the-shelf metal assay of some commercially available "high-purity" metal compounds is compared with the theoretical analytical values in Table 3.

Table 3

Compound	Spectrographic Purity	% Metal Found	% Metal Theoretical
$\text{MnO}_2$	5-9s	62.1	63.19
$\text{Nd}_2\text{O}_3$	3-9s	82.3	85.73
$\text{CdO}$	5-9s	75.3	87.54
$\text{Co}_2\text{O}_3$	5-9s	79.4	71.07
$\text{SrCl}_2 \cdot 6\text{H}_2\text{O}$	5-9s	36.1	32.85

It is clear from the data that high spectrographic purity does not connote high stoichiometric purity.

## PURIFICATION

As an alternative to preparing high-purity inorganic compounds from spectrographically pure metals, one can resort to various purification techniques to reduce the contaminants in reagent-grade chemicals. If the material is soluble in water or an organic solvent, recrystallization is often effective, especially if the impurities are insolubles which are readily filtered out. When the material itself is insoluble, leaching with a suitable acid frequently

leads to selective dissolution of impurities. A good example is the treatment of  $ZrO_2$  with HCl. The iron content can be reduced by approximately an order of magnitude since it is present as  $Fe_2O_3$  which is soluble in HCl while  $ZrO_2$  is not. This technique is of course limited to physical mixtures as opposed to compounds; hence a trace of Si in high-purity  $Al_2O_3$  can be leached out with HF only if present as  $SiO_2$  not as an aluminum silicate.

Selective precipitation can also be accomplished with numerous specific reagents capable of precipitating metallic impurities from solutions.  $H_2PtCl_6$  in dilute HCl can be freed of all but a trace of palladium by adding a solution of dimethylglyoxime which precipitates the palladium as an easily filtered complex. Organic reagents such as dithizone, oxime, and cupferron can readily extract individual trace metals quantitatively from an aqueous solution, into an immiscible organic layer. Heavy metals, particularly, are extractable from alkali and alkaline earth compound solutions which would not react with many organic chelates.

Ion exchange and other chromatographic separations, merit prominent mention. Here ion size, charge and solvent-solute interactions control their selectivity coefficients and advantage can be taken of differences in ion chemistry. Passing a 2M HCl solution through a Dowex 1-X8 anion exchange column will separate iron traces from high-purity nickel salts. Being in a cationic form,  $[Ni(H_2O)_6]^{+2}$ , the nickel ions will pass directly through the column whereas the iron, in anionic form  $[FeCl_4]^-$ , is retained on the resin. Many impurities can be removed simultaneously in this fashion if the anion-cation equilibria of metal ions are considered.

Another method of purification involves volatilization. Halides of many elements such as As, Sb, Sn, Ge, Si, sublime or evaporate readily at elevated temperatures. Distillation of volatile compounds like  $GeCl_4$ ,  $CrOCl_2$  and  $RuO_4$  is also practical for some elements, especially since all-Teflon distillation equipment is now available.

Finally, electrochemical reduction at a mercury cathode removes trace heavy metals from water or aqueous solutions of metal salts. The technique is, limited to those ions which can be reduced to form an amalgam.

## CHARACTERIZATION

After it is produced the high-purity material must be characterized as to purity. The amount of trace cationic, anionic, and neutral impurities as well as stoichiometry are all significant. Even the crystal modification or phase of a material can be crucial for an essential application.

Because of its versatility, optical emission spectroscopy is probably the most ubiquitous tool for the determination of trace cationic impurities. With minimal sample preparation its detection limits for most elements rival other more tedious methods. Although the literature abounds with specialized analytical "recipes" for determining trace impurities, few have widespread applications. A dc arc combined with a Stallwood Jet to reduce background, and high-purity graphite electrodes will generally provide a good start. To further enhance contrast for the faint lines of trace elements, the electrode images are

masked from the entrance slit of the spectrograph during arcing. Whether for semiquantitative or quantitative work Kodak SA#1, 33, or SA#3 spectrographic plates are excellent for most metals. Alkali metals whose strongest lines are at higher wavelengths require red-sensitive 1-N plates.

Powdered samples may be burned directly, alone, or mixed with a series of standards in a suitable matrix—most often graphite. Dilution tends to minimize errors from matrix effects and implement total consumption of such refractory materials as  $Al_2O_3$  or  $TiO_2$ . Likewise, dilution with a lithium carbonate base will enhance lanthanide line intensities. And, borrowing from carrier distillation techniques, high-purity AgCl can be mixed with the refractory to reduce detection limits; upon arcing the more volatile elements (As, B, Pb, etc.) are distilled out of the matrix. If a complex matrix spectrum is undesirable, an additive can be chosen to suppress the spectrum of the major component by suppressing its volatility. In this fashion, with a  $PbCl_2$  carrier and a  $Y_2O_3$  suppressant, 26 trace elements have been determined in  $UF_4$  satisfactorily [6].

Contrast of faint trace impurity lines against background can also be enhanced with a holographically ruled grating. Spectra of a series of Spex G and L standards, taken in the 2350-3500A region in the second order with a 600 g/mm standard ruled grating and in the first order with a 1200 g/mm holographically-ruled grating, revealed that all lines from the holographic grating are real; no spurious ghosts or satellites appear and the general background is fainter by many fold. A lower efficiency of the holographic grating in the UV makes it inferior for the analysis of micro samples. However, increasing the sample size 3 to 5 times to compensate for the loss in speed, we found a dramatic improvement in line-to-background ratios resulting in an average factor of perhaps 3 in detectivity for most trace elements.

For very low levels of trace impurities, fortunately, our bag of tricks includes some concentration techniques: coprecipitation with various reagents, ion exchange, or solvent extraction. All of these procedures depend on extreme cleanliness and ultra-pure reagents. Choice of standards is another weighty consideration.

With care and the Spex seven or five-step standards quantitative analyses have been achieved although semiquantitative suffice. The success of the method depends, however, on several properties of the standards including the blank, the accuracy of the metal concentrations in each and their homogeneity. The blank reflects the purity level of the matrix. Accuracy is assured by preparing standards from assayed metal compounds with multiple-step dilutions and homogeneity is attained by thorough grinding and mixing of very fine particles.

For the very dilute standards imperative for trace-element analyses accomplishing homogeneity can present the knottiest problem. Consider the fact that a 10 ppm standard of PbO in graphite would contain just 30 particles of PbO per gram, assuming 400-mesh spherical particles and an 11.3 g/cc density. What then happens to reproducibility after further dilutions and with even smaller sample sizes? The severity of the problem decreases, of course, with the density of the material when efficient grinding controls the particle size and larger electrodes allow for a healthy, more representative portion of sample.

Also to be considered is the difference in detection limits among the elements. While less than 1 ppm of a few and 5-10 ppm of most cations are observable, some, such as As, P, Ba, W, Th, and U are discernible only under specially contrived circumstances. NaCl, showing no spectral lines but Na, can safely be called 5-9s spectrographic purity, but MoO<sub>3</sub> containing 5-35 ppm W will appear identical with W-free material. It cannot safely be called 5-9s without a further check for W by another method. For some elements like Hg and Cd aqueous atomic absorption offers the needed better detection limits. For others it may take the tedious route of preliminary solvent extractions into organic solvents such as MIBK to raise detectivity several orders of magnitude. The cold vapor technique of flameless atomic absorption is valuable for Hg and elements that form volatile hydrides and with a graphite furnace, picogram quantities of even more elements can be detected.

Alkali metals respond to flame photometric analysis—down to sub-ppm levels in certain matrices. As with flame atomic absorption, the sample must first be dissolved. But UV-visible absorption is often an alternative to flame spectroscopy. After color development with appropriate reagents, the absorbance of the trace cation can be measured against that of a series of standards either in aqueous solution or by concentration into an organic solvent by solvent extraction. Some methods are accurate and specific for trace cations in most matrices, but since the absorption bands are usually rather broad, interferences are quite common. Furthermore, dissolution, color development, and extraction procedures are burdensome and time consuming.

Even lower detection limits can be reached with more elaborate equipment. Being non-destructive, neutron activation is particularly effective for refractory materials. But along with spark-source mass spectroscopy, this sophisticated, expensive instrument system is beyond the scope of most laboratories, including ours. And of similarly limited applicability are electrochemical techniques. Polargraphy and anodic stripping voltametry require dissolved samples and the latter, particularly, is relevant only to easily reduced cations.

Having established the trace cation level in our high purity compound, it is well to determine the amount of anionic impurities. If Cd(NO<sub>3</sub>)<sub>2</sub> is ignited to form CdO and the temperature of calcination is 200C above the decomposition temperature and the reaction is allowed to go to completion, no residual nitrate ions should be present. If, however, PbCl<sub>2</sub> is precipitated from a Pb(NO<sub>3</sub>)<sub>2</sub> solution, then residual nitrate ions could remain in the product. Likewise, if AgBr is precipitated from aqueous AgNO<sub>3</sub> with a stoichiometric amount of HBr, occluded AgCl might appear, since appreciable amounts of HCl are found in reagent HBr. Trace anionic analysis is then dictated by either specific ion electrodes, UV-visible spectrophotometry or nephelometry.

Available for many ions, specific ion electrodes are quite selective for the halides, cyanides, nitrates, perchlorates, sulfides and several less common anions. The potential of each electrode against a reference changes by approximately 59 mV for a singly charged ion and for each

order of magnitude of concentrations; generally levels of 10<sup>-5</sup> to 10<sup>-7</sup> M can be detected, although some electrodes do not do as well and occasionally other anions or metal-anion complex formations interfere. Ever-present is the need to dissolve the sample. Nephelometric measurements comparing transmittances of AgCl or BaSO<sub>4</sub> suspensions with those of standards will provide chloride or sulfate determinations and the trusty spectrophotometer will usually locate sub ppm quantities of arsenate, phosphate, nitrate, nitrite and innumerable other ions [7]. For insoluble materials and well-endowed laboratories neutron activation, x-ray fluorescence or mass spectroscopy are always excellent tools.

Like most anions neutral impurities in high-purity compounds may also go undetected by emission spectrography. Interstitial carbon in iron or dissolved oxygen in silver can be alternatively determined by standard methods to very low levels. Ignition in oxygen in an induction furnace followed by gravimetric or gas chromatographic analysis will also detect carbon, by the resulting CO<sub>2</sub>. Vacuum fusion will spot other dissolved gases including N<sub>2</sub>, O<sub>2</sub> and H<sub>2</sub>.

Among metal oxides, especially those which are very basic, there is a strong tendency to absorb atmospheric moisture or carbon dioxide which reduces the assay of the compound. Moisture content is revealed, however, by loss on drying, CO<sub>2</sub> by loss on ignition and either is detectable by TGA. In many of the metal salts, moisture can be detected by a Karl Fisher titration after treating the compound with anhydrous methanol. Dust or organic residues picked up from filter pads can be determined by dissolving the compound in a suitable solvent, filtering the solution, and weighing the insoluble residue. If SiO<sub>2</sub> or another inorganic is suspected the organic residue can be volatilized by ignition and the inorganics then reweighed.

To further characterize a high-purity material, not only is its level of impurities important, but also its stoichiometry. If the compound is an oxide, an assay of the metal percentage may be sufficient to obtain the empirical formula. If more than two cations are present, however, as in ammonium molybdate, an assay of both % NH<sub>4</sub> and % Mo would distinguish between (NH<sub>4</sub>)<sub>2</sub>MoO<sub>4</sub> and (NH<sub>4</sub>)<sub>2</sub>Mo<sub>7</sub>O<sub>24</sub>•4H<sub>2</sub>O or a mixture of the two. Likewise, to distinguish between BiOCl and BiCl<sub>3</sub> an assay of both % Bi and % Cl and their mole ratio is the best way to establish stoichiometry.

Among many methods of assaying for either cations or anions in a compound are gravimetric procedures. They are amply described in the literature and, done properly, are accurate for many metals and nonmetals. Furthermore, with negligible interferences from impurities, separations may be dispensed with and the precipitate dried at low temperatures to constant weight or ignited at high temperatures to an oxide. Noble metals may be reduced under H<sub>2</sub> and weighed as the metal.

Volumetric techniques may be the most rapid. For alkaline earths and other metals, many transition elements, and the lanthanides, EDTA titrations are close to ideal. The EDTA solution is easily standardized with high-purity zinc or bismuth and error of 1-2 parts per thousand is

typical. For other cations and some anions precipitation titrations or oxidation-reduction titrations such as iodometry are preferable. Instrumental detection of end points further enhances the accuracy of the titration minimizing errors too often less than 1 part per thousand.

In trivalent cobalt, chromium, noble metal complexes or other compounds where isomerization is possible the usual analyses will not distinguish between isomers. In commercial hydrated chromium chloride, for example, there are three readily formed isomers:  $[\text{Cr}(\text{H}_2\text{O})_6]\text{Cl}_3$ ,  $[\text{Co}(\text{H}_2\text{O})_5\text{Cl}]\text{Cl}_2 \cdot \text{H}_2\text{O}$ , and  $\text{trans-}[\text{Cr}(\text{H}_2\text{O})_4\text{Cl}_2]\text{Cl} \cdot 2\text{H}_2\text{O}$ . Since all three isomers contain identical percentages of Cr, Cl and  $\text{H}_2\text{O}$ , an assay will not distinguish among them. But a UV-visible spectrum will; and it will detect the presence of a mixture as well. So will an infrared or Raman spectrum since the coordinated ligands and symmetry of the three isomers are also different,  $\text{Oh}$ ,  $\text{C}_4\text{v}$ , and  $\text{D}_4\text{h}$  respectively.

Mixtures can even appear in a stoichiometric material if more than one phase or crystal type is known. Aluminum oxide can exist as three crystal types,  $\alpha$ ,  $\beta$ , and  $\lambda$ , depending on the temperature and conditions of formation. In this case x-ray diffraction would be the method of choice to determine the phase present.

#### PURE MATERIAL HANDLING TECHNIQUES

Once a high-purity material has been synthesized and characterized, its quality must be maintained by careful consideration to handling, grinding procedures, and storage containers. Almost any grinding equipment will introduce some impurities through abrasion but generally the harder the container material the less the contamination. Boron carbide, silicon carbide, and tungsten carbide are high on the Knoop hardness list. Plastics should not even be mentioned in the same sentence. But they do not introduce spectrographically detectable elements.

In one experiment a 10g sample of spectrographic grade  $\text{Li}_2\text{CO}_3$  was ground for 2 minutes in a hardened steel dish, a tungsten carbide dish, and in a polypropylene vial with a lucite ball. A spectrographic analysis of the material after

grinding revealed a large amount of iron (500-1000 ppm) in the material ground in steel, small amounts of tungsten and cobalt (50 ppm) and less iron and nickel (20 ppm) in the material ground in tungsten carbide, and no inorganic impurities in the material ground in plastic. A subsequent determination of the amount of plastic residue in the  $\text{Li}_2\text{CO}_3$  revealed approximately 400 ppm. This material could of course be removed by washing with an organic solvent or by ignition if necessary. In another experiment several vials of the same material were shaken for 5 minutes with 1g of graphite and 1g of 6-9s  $\text{SiO}_2$ . An analysis of the powder after mixing showed a trace of calcium (<1 ppm) as the only contaminant picked up from the abraded vial.

Clear plastic containers are generally good for storing high-purity chemicals provided they are not light sensitive or corrosive. In addition to the admonitions already mentioned in our discussion of reaction vessels, air oxidation, and moisture absorbance must be considered.

**A**LTHOUGH it has been our intent here to summarize most of the major considerations in the preparation of high-purity materials, by no means is it meant to imply that all high-purity compounds must be fully characterized by the most extensive battery of tests for every application. The term "high purity" remains very relative; for any given application it is far better to have more information than required than not enough.

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May we send you a price list of materials typically supplied or quote your special mixture or unit weight for your requirement? We'll work with your material or ours.

Our precision automatic balance is faster, cheaper, more accurate and probably more enthusiastic about weighing diluents than your lab technician!

Individual chemicals or any mixture you specify are loaded to a tolerance of  $\pm 2$  mg into plastic or glass containers ready for the addition of your sample and popping into a Spex Mixer/Mill or Wig-L-Bug.

SPEX Preweighs  
can take you  
out of the

ICE AGE

# NOW YOU CAN SPECIFY YOUR OWN SEMIQUANTITATIVE STANDARDS

Perhaps you have relied on our semiquant standards for as long as you've been untangling unpredictable unknowns. But did you know that we have recently been preparing special standards for special needs? They include:

BASE	ELEMENTS	RANGE, %
Graphite	49 common	0.1-0.0001 in 9 log steps
Graphite	10 common	0.01
Graphite	Rare earths	0.5-0.005 in 4 log steps
Graphite	9 common	0.005-0.0003 in 4 lin steps
MoO <sub>3</sub>	19 common	0.064-0.0005 in 9 lin steps
Al <sub>2</sub> O <sub>3</sub>	5 common	0.05-0.9 in 5 steps
Graphite	Te, Cr, Cu, Mo, Co	1% each
Graphite	Te, Rb, Zn, Cr, Ca	1%
Al <sub>2</sub> O <sub>3</sub>	Mn, V, Cr, Zn, P	0.1-0.001 in 3 log steps
NaCl	28 common	0.1-0.0001 in 7 linear steps

And for AA Authorities here are a few of our special solution standards:

BASE	ELEMENTS, ppm
Methylisobutylketone	V, 1000
Methylisobutylketone	Sb, 1000
5% HF	Ti, 5000 + Al, Fe, Sn, V, 1000
2% KCN	Au, 171.4
2% HCl	Al 6-9s pure, 10,000
20% HCl	Au, 5000 + 100 ppm of 10 other elements

## TIME-SAVER STANDARDS

Despite their 20-year-old reputation for being able to analyze anything semiquantitatively, our graphite-base G Standards do have their limitations. If you wish to reference an unknown silicon sample against the G Standards, the sample must first be diluted 10-100 times with graphite powder. Not unexpectedly, detectivity plunges by the same amount. Wouldn't a set of reference standards based on silicon be ideal?

That's exactly the idea behind Spex TIME SAVERS. You choose the base and, further, you choose the elements you wish to determine. They can be a SPEX MIX containing 49 common elements, or 16 rare earths, or 10 noble metals, all at the same concentration.

At present, we stock a number of 6-9s-pure base materials. From these a 5-part set of TIME-SAVER standards can be doped at 0.1%, 0.033%, 0.01%, 0.0033%, and 0.001%. Since the base material itself harbors less than 1 ppm of total impurities, the standards will be applicable to every element in the SPEX MIX without need for blank corrections.

It's important to note, however, that often lesser purity base materials are suitable. Our 5-9s WO<sub>3</sub> is relegated to second-class citizenry only because of its intimacy with one element, K, at around 5-10 ppm. Similarly, third-class status is imposed on Ta<sub>2</sub>O<sub>5</sub> because Nb<sub>2</sub>O<sub>5</sub> is such a tenacious companion at levels of 10-50 ppm.

So, if you are working with materials which we supply at the 6-9s level, you can ignore base contaminants. If, on the other hand, you require one of the base materials in the 5-9s or 4-9s groupings check with us (attn: Warren Miller) before concluding that its purity will not do. Its semiquantitation may well be suited to your needs.

**TS 4 TIME-SAVER STANDARDS**, set of 5 plus base for "blank" measurements; 2g each; Specify SPEX MIX diluent #1000 Common Elements; #1031 Rare Earths; or #1041 Noble Metals; and 4-9s pure base:

CERIUM DIOXIDE, NIOBIUM PENTOXIDE, TANTALUM PENTOXIDE,	URANIUM TRIOXIDE, ZIRCONIUM DIOXIDE,	set \$85.00
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**TS 5 TIME-SAVER STANDARDS**, set of 5 plus base for "blank" measurements; 2g each; Specify SPEX MIX diluent #1000 Common Elements; #1031 Rare Earths; or #1041 Noble Metals and 5-9s pure base:

ANTIMONY TRIOXIDE, ARSENIC TRIOXIDE, BARIUM CARBONATE, BISMUTH TRIOXIDE, BORON OXIDE, CADMIUM OXIDE, CALCIUM CARBONATE, CHROMIUM SESQUIOXIDE, COBALTIC OXIDE, CUPRIC OXIDE, FERRIC OXIDE, LANTHANUM OXIDE, LEAD MONOXIDE, MAGNESIUM OXIDE,	MANGANESE DIOXIDE, MERCURIC OXIDE, MOLYBDENUM TRIOXIDE, NICKEL OXIDE, POTASSIUM CARBONATE, SELENIUM DIOXIDE, SODIUM CARBONATE, STRONTIUM CARBONATE, TELLURIUM DIOXIDE, THALLIC OXIDE, TITANIUM DIOXIDE, TUNGSTEN TRIOXIDE, or VANADIUM PENTOXIDE,
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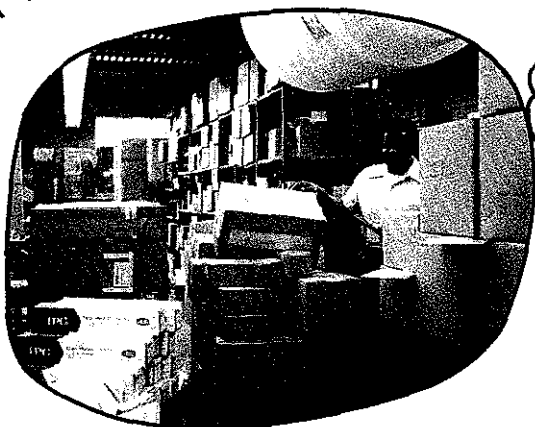
set \$95.00

**TS 6 TIME-SAVER STANDARDS**, set of 5 plus base for "blank" measurements; 2g each; Specify SPEX MIX diluent #1000 Common Elements; #1031 Rare Earths; or #1041 Noble Metals and 6-9s pure base:

ALUMINUM OXIDE, GALLIUM OXIDE, GERMANIUM DIOXIDE, INDIUM OXIDE,	SILICON, SILICON DIOXIDE, TIN DIOXIDE, or YTTRIUM OXIDE	set \$120.00
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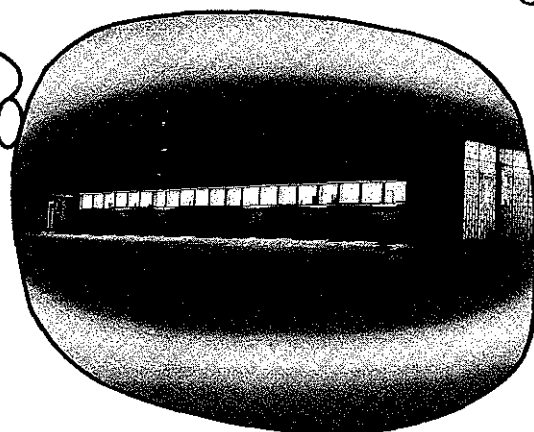
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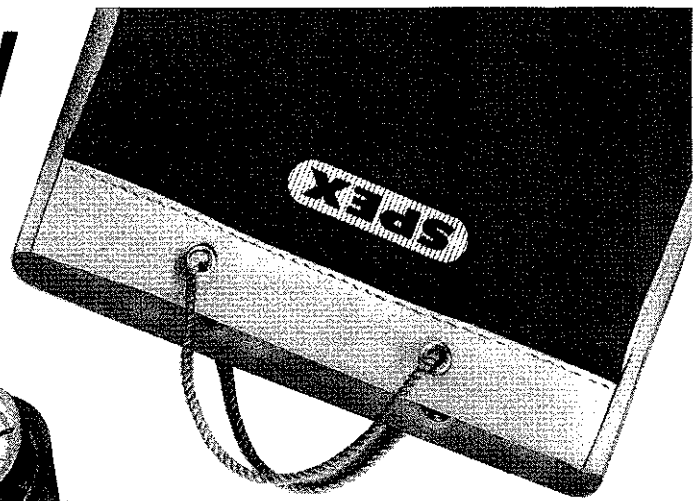
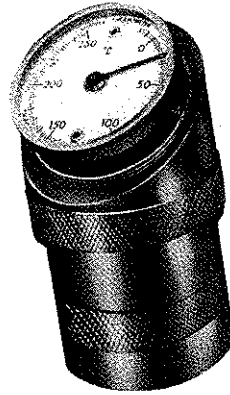
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