ICAP DIAGNOSTIC TEST

The purpose of this test is to determine if the spectrometer is properly working and to isolate the problem area if it is not. It is designed to be run unattended over a relatively long term (four hours) and therefore is convenient for overnight execution.

SOLUTIONS NEEDED

1. Blank: 10% HCL (1000 ml)

2. Test STD: 10% HCl plus 10 ppm As, P, Cu, Pb, Ca; 1 ppm Ba, Cd and Mg (1000 ml)

It would be best if you use TJA standards specifically prepared for this test, part number: (to be created) (it is a set that includes standard and blank, 1 liter ea.). These standards are properly preserved and can be used for at least 6 month after opening. The standard might have more elements than listed above. It would not affect the test.

METHOD

Develop a method with the following lines;

As1890	Ba4934	
Cu3247	Cd2265	P1782
Ca3179	Mg2852	Pb2203

These lines will give you a cross-section of lines which are visible, UV, RF sensitive, and nebulization sensitive. Set up the method with the above stated concentrations for the elements in the test standard and select background correction

points for each line (for sequential units use “automatic” bkg correction option, for Iris - use default positions).

Complete methods development as appropriate selecting the recommended plasma parameters:

RF power - 1150, aux. flow - low, pump rate - 100 rpm (constant for flush and analysis), neb. pressure - should be optimized for the employed nebulizer(see attached).

For sequential units it is important to put all elements in the same plasma group.

Integration time should be 2 sec for all elements for sequential ICP. For Iris units with standard resolution (radial or axial) integration time should be 30 sec for the first slit and 5 sec for the second slit. For Iris units with high resolution (radial or axial) integration time should be 40 sec for the first slit and 20 sec for the second slit.

Use at least three replicates.

STANDARDIZE THE SYSTEM

1. prior to standardization:

Be sure to perform Hg calibration and PEAK SEARCH if testing a sequential.

Be sure to verify that all subbarays are centered when testing an Iris.

Be sure to profile a polychromator when testing 61E, Trace, et.

2. Use the blank and test standard to **standardize** the spectrometer after it has been properly warmed up (at least 30 minutes after the plasma was turned on).

If you are running an axial instrument that has an internal standard kit connected, please replace it with a single orange-orange pump winding for this test.

3. Examine RSDs on the high standard (NOT the blank).

If you have cyclonic glass spray chamber with Meinhard nebulizer RSDs on the high standard should be no worse than 1 % for simultaneous instruments and 2 % for sequentials.

If you have barrel glass (with baffles, Scott style) spray chamber with crossflow nebulizer RSDs on the high standard should be no worse than 1.5 % for simultaneous instruments and 3 % for sequentials.

If you have cyclonic glass spray chamber with Burguner nebulizer RSDs on the high standard should be no worse than 1.5 % for simultaneous instruments and 3% for sequentials.

If you have any special configuration of sample introduction system (HF resistant, water cooled, et., precision may vary depending on configuration).

If obtained RSDs are within these guidelines, you can proceed with the test. If RSDs are significantly higher, you should locate the source of poor precision and fix that problem prior to moving to the next step (check nebulizer for clogging. argon leaks, vent flow, et.)

4. After standardization please run the blank solution as an unknown (7 replicates for each run, run at least twice) and multiply the data by a factor of 3 to determine an **instrument detection limit** of the system.

COMMAND FILE

For ThermoSPEC DOS, create the following command file;

AM+ # initialize auto mode

{20UAZ WM10} # analyze an unknown 20 times with a 10 min. wait in between

TOR- # shut down the torch at the end

It should be written the following way in the "Command" line in analysis:

am+ {20uazwm10} tor-

Prior to command execution the method should be set up to autostore the data (for Iris instruments it would be helpful to store image data as well).

For ThermoSPEC WIN, the same command file can be used except eliminate the first command, AM+. The wait time between samples can be adjusted to whatever you prefer; W30 for example will wait 30 seconds; WM5 will wait 5 minutes, etc.

RUN IT

Make sure you have enough solution for the time designated in the test.

Put the sipper probe in the test standard and initiate the command file.

INTERPRETATION OF THE TEST

The lines selected have the following significance:

1. As1890 and P1782: UV sensitive lines; if only these two lines behave poorly, i.e. high RSD's and drift, optical coating or poor purge may be indicated.

2. Cu3247 and Ba4934: Sample introduction sensitive lines; look for problems with the pump windings, nebulizer, spray chamber, center tube, or nebulizer pressure. These lines are also higher wavelength lines which suffer more from stray light problems. On all IRIS's, the Ba line is on the off-axis slit; the Cu line may be on the off-axis slit. This may be significant.

3. Cd2265, Ca3179, and Pb2203: RF sensitive lines; could be indicative of a faulty or worn power amplifier tube.

The drift specifications are: 2% per hour for simultaneous instruments and 3 % per hour for sequentials (if intensities and precision look fine).

Instrument detection limit is also very important for ICP performance evaluation. It was calculated prior to the stability as 3 times the STD (standard deviation) of the blank. To evaluate your current detection limit please compare it to the value in the "blue book" (original set of data that came with the instrument from the factory). If the detection limits are more than a factor of 2 higher than the "blue book" value, you might need to reoptimize the system.