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應用手冊

AutoTune and Resolution Checker - New Automated Functionality for AutoSpec - *Ultima* NT

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This is an Application Brief and does not contain a detailed Experimental section.

Abstract

This application brief describes two of the major new automated facilities available for the Autospec-*Ultima* NT.

Benefits

- The implementation of AutoTune for AutoSpec-*Ultima* NT allows inexperienced and experienced users to rapidly obtain the required resolution at high sensitivity
- The Resolution Checker for AutoSpec-*Ultima* NT ensures that results are validated and regulatory requirements are met

Introduction

This brief describes two of the major new automated facilities available for the Autospec-*Ultima* NT.

The AutoTune facility has been developed as a time saving tool to assist both inexperienced and experienced instrument users. A sophisticated tuning algorithm combined with the latest in automated ion optics can reliably produce the desired resolution with good sensitivity.

The Resolution Checker can be automatically deployed between samples, or at any given point in a sample sequence, to verify that the experimental resolution is within specified tolerances. The Resolution Checker has the capability of halting the sample list if the resolution is out of tolerance, preventing the loss of valuable samples. With its automatic printing of reports, Resolution Checker is a powerful validation tool.

Results and Discussion

AutoTune

Prior to the implementation of AutoTune, the Tune Optimize facility provided the functionality of just a simple re-optimization of just a few of the analyzer lenses. AutoTune takes automated instrument tuning to a new, comprehensive level. The AutoTune facility is designed to achieve a resolution within a specified tolerance by finding the optimum setting for the lenses and the source and collector slits. This tolerance is set by the operator. The AutoTune program can be set to achieve any resolution between 1000 and 15000 (10% valley definition) from any starting resolution where a peak is visible in the Tune window. Figure 1 shows the Tune window, Status dialog and Parameter dialog after an AutoTune to 10000 resolution with a specified tolerance of +5% and –0%.

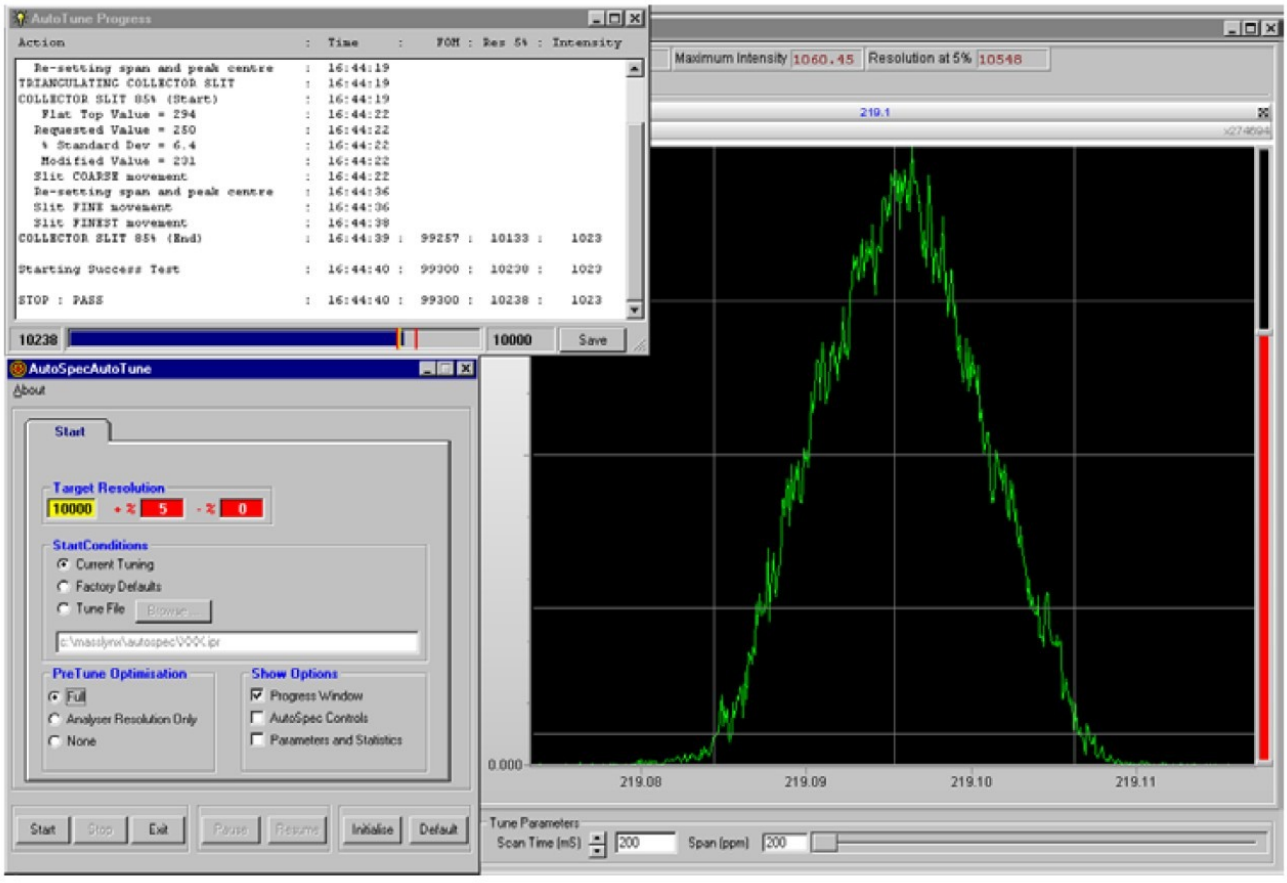


Figure 1. Tune window, Parameter dialog, and Status dialog for AutoTune.

For a magnetic sector instrument the ultimate resolution is limited by the width of the

source slit, which affects the number of ions entering the analyzer region of the instrument. Attaining the best sensitivity for a given resolution is achieved by optimizing the setting of the source slit.

The AutoTune facility will measure the current resolution and intensity and then adjust the source slit to a width calculated to give the best transmission of ions for the desired resolution. Subsequent to setting the source slit width, the AutoTune program optimizes all the lenses of the AutoSpec- *Ultima* NT using iterative procedures if required.

An option is available for the AutoTune program to perform a 'Pre-Tune' of the lenses prior to the adjustment of the source slit. This would be appropriate to the situation where lenses and slits have been set randomly and not tuned. Although this is an extra step, it improves the reliability of the AutoTune process, as this will set the lenses nearer to their final optimized setting prior to calculating the source slit width. This increases the likelihood that the desired resolution will be achieved with a single setting of the source slit.

AutoTune Performance

Success Rate

Two sets of experiments were carried out to test the success rate of the algorithm. The algorithm was deemed to be successful if the achieved resolution fell within the tolerance specified. In this case the resolution tolerance was set to + and – 5%.

Firstly the AutoTune was set to tune to a large number of randomly generated resolution values between 1000 and 15000. After the first tune each subsequent tune started from the tuning conditions of the last successful AutoTune procedure. This ensures that the program was tested whilst targeting both higher and lower resolutions than the current resolution. Secondly, the AutoTune program was set to target the same range of resolutions but starting from random tuning conditions. In these cases a Pre-Tune is performed. The results of the experiment are summarized in table 1.

For the first set of experiments the success rate was 99%. For the second set of experiments, where initial tuning conditions were randomized and the 'Pre-Tune' was used, the success rate was 93%.

The average duration of the successful AutoTune procedures were:

1. Without a Pre-Tune : 5 ½ mins

2. With randomized tuning conditions and a Pre-Tune : 11½ mins

	Number	Percentage
Total Number of Tunes from good initial tuning conditions (Pre-Tune not used) :	480	
Number that Passed :	475	99%
Total Number of Tunes from random initial tuning conditions (Pre-Tune used) :	564	
Number that Passed :	524	93%

Table 1. Summary of results of assessing the success rate.

AutoTune vs Manual Tuning

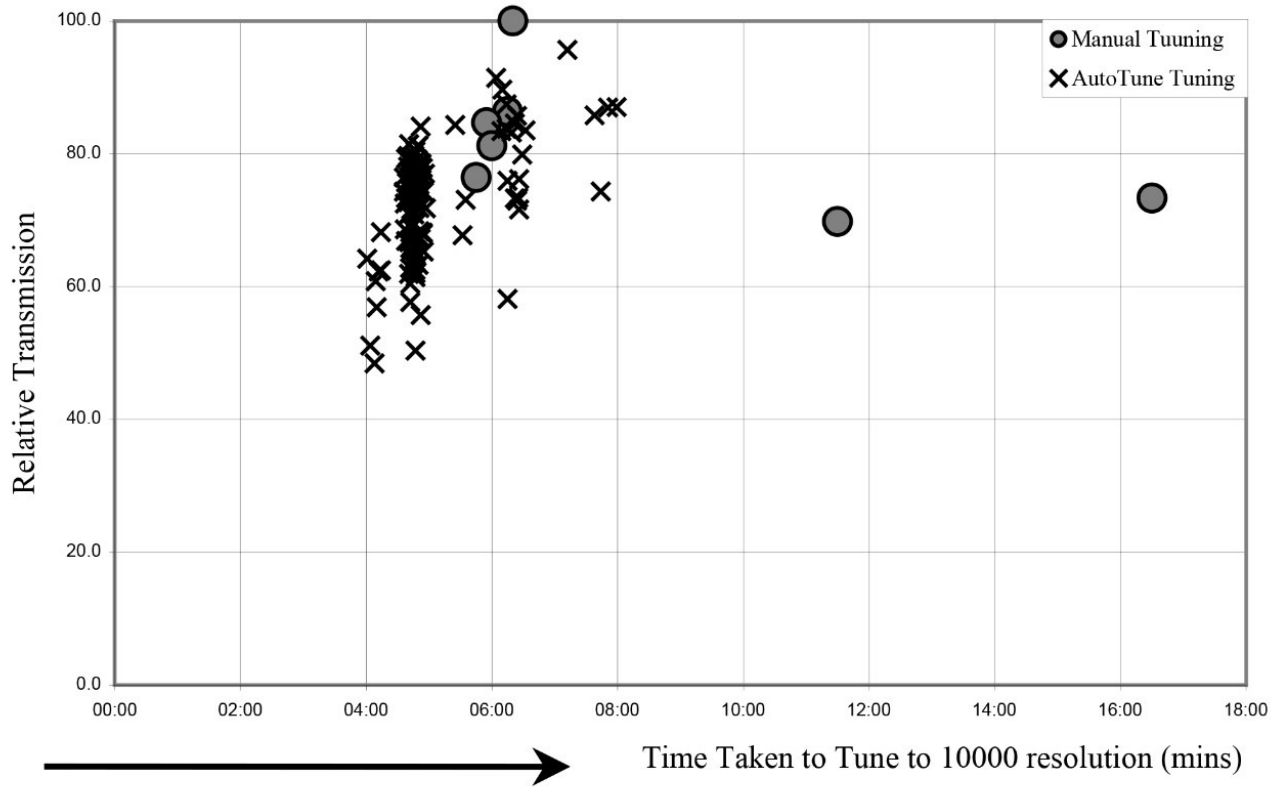
To compare the performance of AutoTune against users with a range of instrument experience an experiment was conducte whereby operators were asked to tune from a 1000 resolution to 10000 resolution. The AutoTune program was then repeatedly deployed to target the same resolution from the same tuning conditions with 'Pre-Tune' not selected.

This experiment was an assessment of time and sensitivity, of which the operators were made aware. The operators participating in this experiment had a range of experience with the AutoSpec-*Ultima* NT (or similar instruments), from 1 to 13 years.

The results of this experiment can be seen in graph 1.

The AutoTune program attempted the tune 122 times with a 100% success rate.

The average time taken for the tune was 5 minutes and 5 seconds.



Graph 1. Summary of results of comparing manual and automated tuning.

The final intensity achieved at 10000 R.P. by AutoTune was on average 89% of that achieved by manual tuning.

Manual tuning produced marginally better results than the AutoTune program, which was to be expected from experienced users. It can be seen, however, that the AutoTune yielded better or at least as good results as several of the operators *i.e.* those who were at the lower end of the experience range.

There was no doubt that the AutoTune program was, in the vast majority of cases, substantially quicker than the manual tuning. This highlights the advantages of AutoTune for the inexperienced user and also how the AutoTune program can increase productivity for experienced users by being used to attain good tuning conditions whilst unattended.

Resolution Checker

Resolution Checker can be added as a process option to any sample in the sample list. To use Resolution Checker it is necessary to first generate a configuration file, specifying which experiment is being used, which reference masses are to be checked, what the resolution tolerances are and whether a hard copy report is required.

When Resolution Checker is deployed it will lock onto the reference peak and subsequently average a number of scans to derive the mean resolution of the peak. In a multifunction experiment, either just the first function or all the functions can be analysed in this fashion. If there is a necessity for the program to run within strict time constraints, for example if Resolution Checker is to be run in the time the GC takes to reset to starting conditions, just the first and last reference masses within the function can be checked.

Subsequent to verifying the resolution of the experiment, Resolution Checker can print out a report, providing hard-copy evidence of the resolution every reference mass checked. An example report is shown in Figure 2. This functionality helps to satisfy the stringent conditions of regulatory analytical protocols such as US EPA Method 1613.

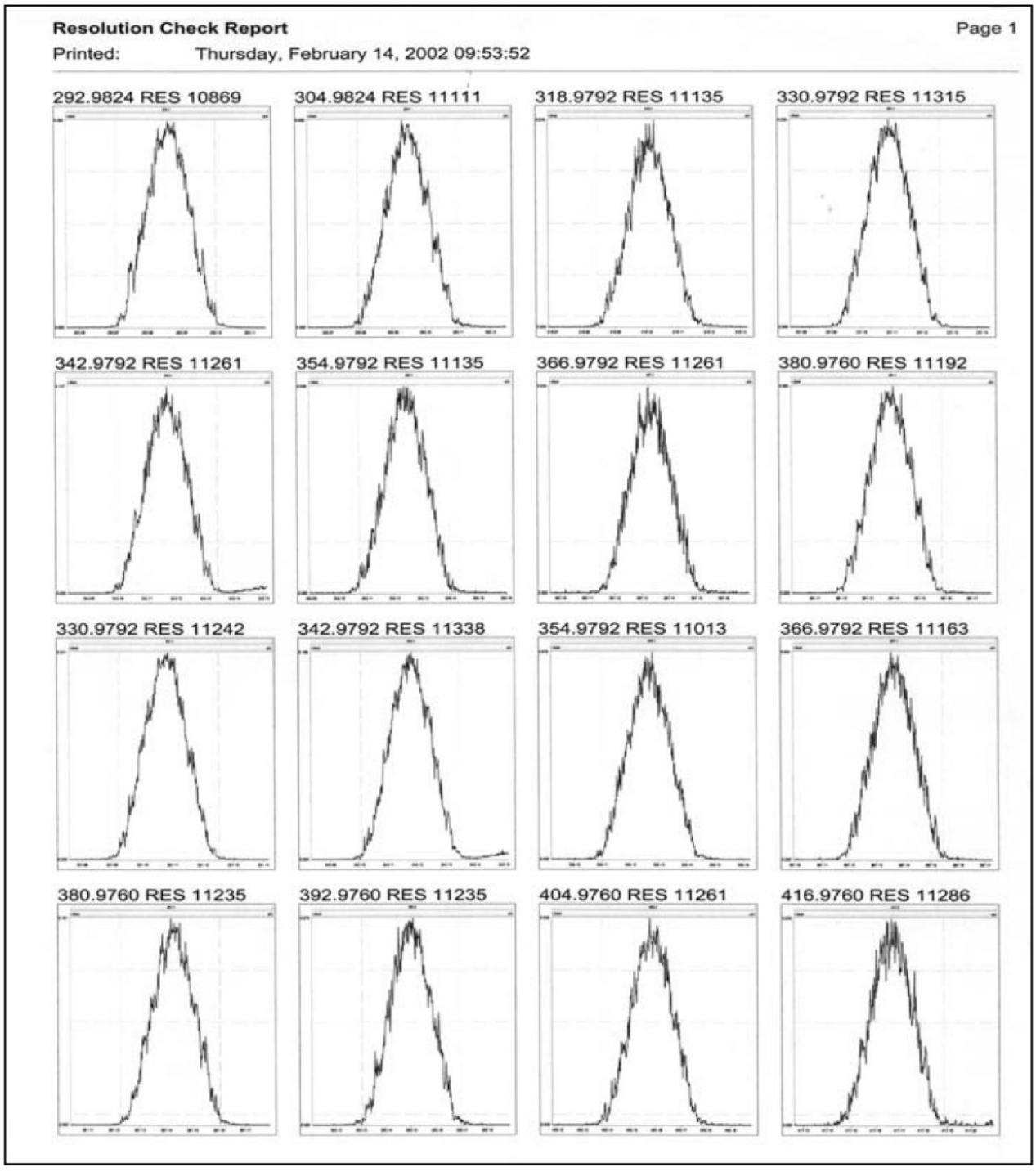


Figure 2. Example of a Resolution Checker report.

Conclusion

The implementation of AutoTune for AutoSpec-*Ultima* NT allows inexperienced and experienced users to rapidly obtain the required resolution at high sensitivity. AutoTune has been shown to be able to tune to high resolution at high sensitivity, similar to that that may be achieved by an experienced user.

Documented resolution checks are a requirement of several regulated methods. The Resolution Checker for AutoSpec-*Ultima* NT ensures that results are validated and regulatory requirements are met.

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