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IMS R&D PROGRAM AT CANADA CUSTOMS.

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ABSTRACT

Over the last few years, Revenue Canada, in collaboration with Barringer Instruments Limited, has been involved in the development of a field-usable ion mobility spectrometer (IMS) for the detection of drugs of abuse. This work has culminated in the manufacturing and commercialization by Barringer of the Ionscan 350 instruments, now in use by various law enforcement agencies worldwide.

Although IMS exhibits a very strong and distinctive response toward some nitrogen containing drugs, e.g., cocaine, like all separation techniques it has inherent limitations, namely moderate resolution and low chemical signal to noise ratio which may affect the reliability of IMS-based drug detectors. A programme is in place at the Laboratory and Scientific Services Directorate (LSSD) to investigate the applicability of various digital signal processing (DSP) techniques to IMS output signals. The application of neural network techniques to overlapping IMS peaks is presented.

INTRODUCTION

The Research and Development Division of the Laboratory and Scientific Services Directorate (LSSD) of Revenue Canada, Customs, Excise and Taxation, has a mandate to perform the following tasks:

- identify and develop technology and instrumentation for the detection of drugs to be used by Customs officers at points of entry into Canada; and

- develop new methods for the Customs Laboratory and the Excise Laboratory.

The work described in this paper gives a brief review of the work performed at LSSD on the development of a drug detection system based on IMS, outlines possible applications of IMS for the Customs or Excise Laboratories, describes modifications performed on the drug detector for other applications and gives details on digital signal processing which may be useful in all applications of ion mobility spectrometry.

Drug Detection

Between 1983 and 1991, LSSD was involved in the development and testing of the IONSCAN series of instruments for the detection of cocaine and heroin in Customs scenarios, in conjunction with Barringer Research Limited (BRL)[1-7]. In 1993 and 1994, Revenue Canada purchased four IONSCAN instruments from BRL, one used for on-going testing in the laboratory and three for field implementation. Two of the field instruments are presently in use at airports in Toronto and

Montreal; these have been instrumental in a number of drug seizures.

Customs, Excise Applications

The Customs Laboratory of Revenue Canada is responsible for the analysis of imported goods into Canada for tariff classification while the Excise Laboratory is involved in the analysis of alcohol and tobacco products for the determination of excise tax. The R&D Division has worked in conjunction with these laboratories to develop methods using ion mobility spectrometry. The samples chosen for analysis by IMS met at least one of the following requirements:

- the sample consists of an analyte of interest present in a well known matrix which is relatively inert to the IMS detector;
- the existing method of analysis for the sample involves a long preparation step; and
- no satisfactory analytical techniques are available at our laboratory.

Some preliminary work on a BRL Ionscan 250 instrument has indicated that IMS can be used for the determination of the presence of additives in polymers, and the presence of cocaine and/or heroin in drug seizures. In addition, IMS can be used to analyze bitrex in denatured alcohol.

RESULTS AND DISCUSSION

Modifications to Ionscan 250.

The Ionscan 250 has been developed for the sampling and analysis of solid samples collected in the field. The instrument uses pumped, purified room air for the drift and carrier gas. An exhaust pump evacuates the drift and carrier gas to avoid the creation of a vacuum or pressurization inside the drift tube. The software of the Ionscan 250 gives an indication of the presence of a substance of interest by monitoring a number of windows across the spectrum. In order to use the BRL Ionscan 250 instrument for laboratory applications, a number of modifications were required. These modifications were performed in conjunction with BRL.

The modified instrument uses zero air from a cylinder and does not require pumps for the drift or carrier gas. The exhaust pump from the Ionscan 250 is still used. Zero air is also used as a make-up gas.

For a more reproducible introduction of samples as solutions, the inlet of the Ionscan 250 was modified, as shown in Figure 1. When no sample tube is inserted into the inlet, a make-up gas is introduced into the IMS; the rate is adjusted so that 15 to 20 cm^3/min of gas come out of the inlet, thus not allowing unpurified room air to enter the drift tube. The sample is injected into the glass wool of a glass sample tube, and the tube is brought into the inlet of the instrument, close to the repeller grid of the drift tube to ensure good transfer of the sample to the reactant region. The carrier gas is set at 200 cm^3/min and is pumped out of the drift tube, along with the drift gas, set at 300 cm^3/min, by the exhaust pump. The make-up gas is flushed out of the front end of the inlet since no tight seal is made between the sample tube and the inlet. The BRL temperature controls were used for the new inlet.

A data acquisition system was developed to increase the flexibility of the instrument. The software has a variable acquisition rate (20 to 125 KHz), allows for a maximum of 512 data point

per scan, has a 30 msec gate firing, a 4 msec delay time for data processing, hand shaking and data transfer. It has the capability of storing individual raw scans and displaying 128 individual traces. 4 megabytes of memory are available for data storage. Figure 2 shows a partial display of 16 traces, chosen from the full display to investigate certain features in the spectrum. From the partial display, single traces can be plotted, where the drift time and amplitude information can be obtained.

Signal Processing

The output signal of the spectrometer is made up peaks embedded in noise, each peak indicating the presence of a specific component in a mixture. Previous analyses of experimental data[8] have shown the following:

- the peak shapes of IMS signals are Gaussian; and
- the noise is band limited, shows no clear repetitive pattern and is Gaussian.

From this information, an IMS signal simulator was developed to artificially generate IMS-like signals with any desired peak parameters (amplitude and standard deviation (σ)), peak separation and signal to noise ratio (SNR)[8]. The simulated signals were subsequently used to test the detection limits and the selectivity of peak detection algorithms. The detection limit of an algorithm is measured by determining how well a low signal can be detected in the presence of noise, without false detection. The selectivity is determined by the minimum peak separation that can be correctly detected by the algorithm. The resolution is a function of the peak separation, the standard deviation of the peak, the relative amplitude and the SNR. A number of packages have been tested previously[8,9]; the results obtained are summarized below. Finally, recent results obtained with neural networks are described.

Derivative Methods

This involves the differentiation of the IMS signal. Any slight variation in the slope of the original signal due to overlapping peaks can be detected using this technique. However, because of this capability to recognize slight variations, this method is susceptible to noise. It must therefore be preceded by precise filtering to remove out of band noise. The results obtained on simulated signals are the following:

- in a high signal to noise environment, with a 1:1 signal ratio, a differentiation of peaks separated by 1.7 σ can be achieved using double differentiation;
- with an amplitude ratio of 6:1, the minimum separation must be 2.2 σ;
- single peaks can be detected at 18 dB SNR;
- single peaks could be detected at lower SNR at the expense of selectivity; and
- the detection of multiple peaks can be achieved at lower separation by using higher order differentiation (1.4 σ using 6th order differentiation, 1.25 σ using 10th order differentiation), at the expense of high false detection probabilities.

Cross-Correlation Method

This method involves cross-correlating two vectors of length n (an IMS signal and a Gaussian curve) for relative shifts of -n up to +n. When the two vectors are normalized, the cross-correlation function is a maximum when the two signals are identical and is zero when the two vectors are uncorrelated. This method is very efficient for the detection of single peaks in low SNR environments and may therefore improve the detection limit of an IMS system. The method has a negative impact on selectivity since it is a perfect filter, blocking all other noises, including irregularities due to the presence of other peaks.

Curve Fitting Methods

In this method, the signal is resolved into distinct bands which are fitted to an n-order polynomial using least squares. The polynomial coefficients are then used to estimate the parameters of the peak. In IMS, a quadratic equation can be used since the peaks are Gaussian. This kind of algorithm is very accurate at estimating the position of a peak.

Hopfield Neural Network

An IMS signal can be thought of in terms of the following equation:

$$r(t) = x(t) + z(t)$$

where x(t) is the signal of interest, assumed to be made up of Gaussian pulses and z(t) is a noise term. The Gaussian pulses can be represented by:

$$g(t;l,k,m) = a_l \exp \frac{-(t-c_k)^2}{2\sigma_m^2}$$

$\{a_l\}$ is a set of discrete amplitudes
$\{c_k\}$ is a set of discrete centers
$\{\sigma_m\}$ is a set of standard deviations

x(t) can then be represented by

$$x(t) = \Sigma \Sigma \Sigma A_{lkm}\, g(t;l,k,m)$$

where A_{lkm} is 1 if g(t;l,k,m) is present in the signal and A_{lkm} is 0 otherwise.

Our objective is to obtain the values of $\{A_{lkm}\}$ from the noisy signal. This is a set of cross-coupled equations for which it is hard to obtain analytical solutions. Thus, it was decided to use a neural network approach to find the solution iteratively.

This problem can be thought of as a multi-input multi-output process where the input is the N-dimensional recorded signal and the output is a binary vector. The system structure is very similar to a well known neural network structure called the Hopfield net. The network consists of a single layer of Q neurons. Each neuron adds up all its inputs and compares the sum to a threshold value. If the sum > threshold, the output of the neuron is 1, if the sum < threshold, the output is 0.

Each neuron output is fed back to the inputs of all neurons except its own. Feedback connections are called weights. The neuron also receives signals from an external source. The weights are determined by minimizing an artificial energy function which is a measure of how far the value of the output vector is from an acceptable solution. The setup of the Hopfield Network is shown in Figure 3.

In order to test the network's sensitivity to noise and its resolving power, simulation studies were performed using an input vector consisting of 1024 points, made up of Gaussian pulses with different amplitudes, centers and sigma, with different SNR. Signals collected on the modified Ionscan unit described above were also used as input into the network. The vector (simulated signal or actual IMS spectra) was fed into the neural network and compared to a set of 12 basis functions, Gaussian functions with varying distances between two consecutive functions and varying standard deviations (see Figure 4). The network calculates the error between the signal and the basis functions with different parameters and displays the best fit between the input and the basis functions.

The system's sensitivity to noise was tested by creating one Gaussian pulse using the signal simulator. The noise level of the signal was increased until the network failed to produce the correct output. The minimum SNR was found to be -3dB, as shown in Figure 5. At this level, the noise power is twice the signal power. Reducing SNR below this level resulted in multiple peaks, as shown in Figure 6 for a blank sample introduced into the modified Ionscan.

The network's resolving power was tested by analyzing two pulses which are synchronized to the positions of their basis Gaussian pulses counterparts. These pulses were delayed by one standard deviation. At equal amplitudes, two peaks were observed for a signal to noise ratio as low as 3 dB (Figure 7). The amplitude of one pulse was then reduced gradually until the network failed to produce the correct output. The limit of amplitude ratio was approximately 0.6 with a signal to noise ratio down to 10 dB. The time separation between two peaks was then reduced. A time resolution of 0.65 σ could be obtained, at a SNR of 30 dB, as shown in Figure 8. Tests on various combinations of delay and amplitude differences indicate that the limit is 0.65 σ at an amplitude ratio of 0.8. An example of the output of the Hopfield Network for the separation of cocaine and tetracaine, injected as a mixture in solution, is shown in Figure 9. These two substances have amplitude maxima separated by approximately 0.65 σ.

We are presently investigating the capabilities of the Hopfield Network in more details, along with the possibility of using a combination of algorithms to lower the detection limits, and to help in the peak location and peak resolution capabilities of ion mobility spectrometers.

ACKNOWLEDGMENT

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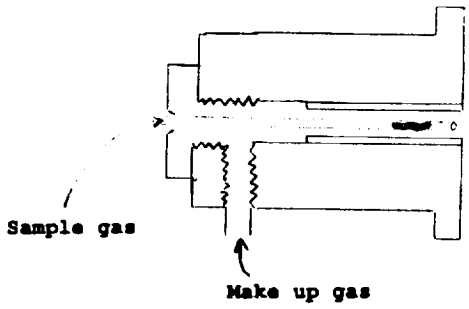

Figure 1. Modified Ionscan Inlet.

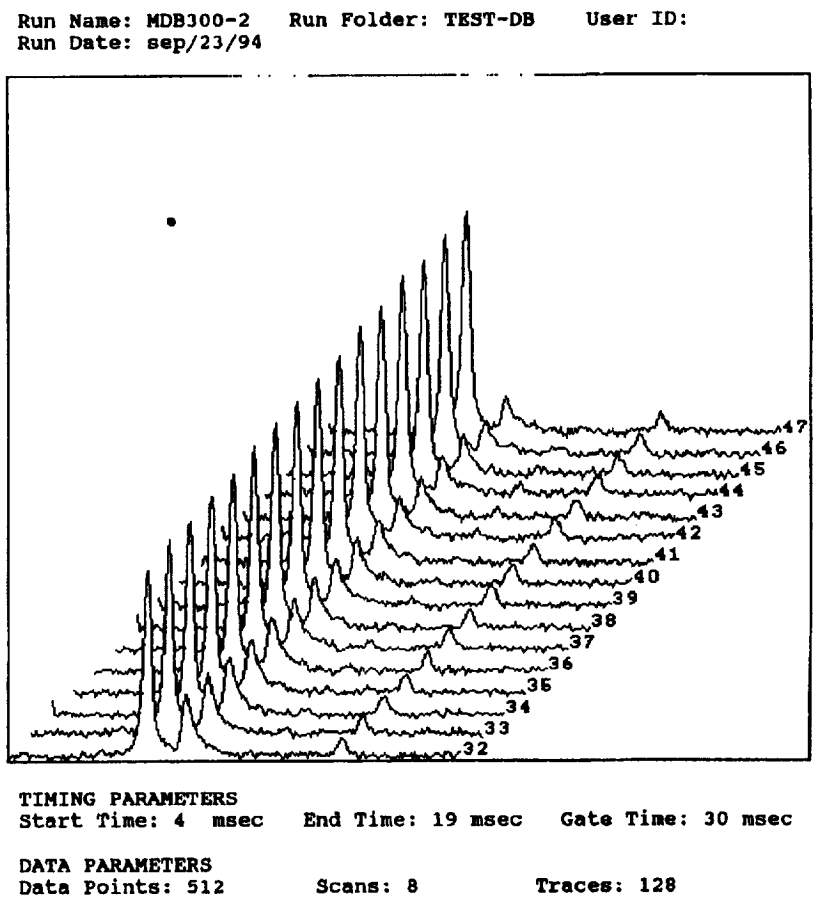

Figure 2. Modified Data Acquisition System: Partial Display

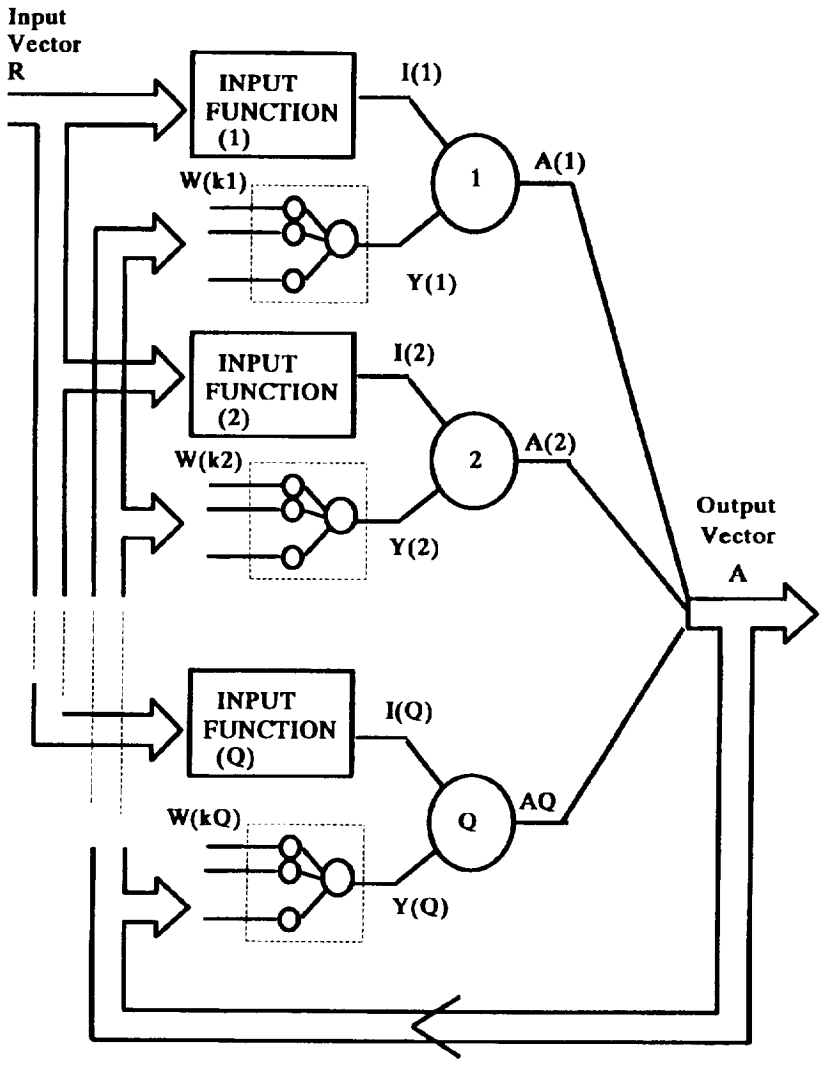

Figure 3. Setup of a Hopfield Network

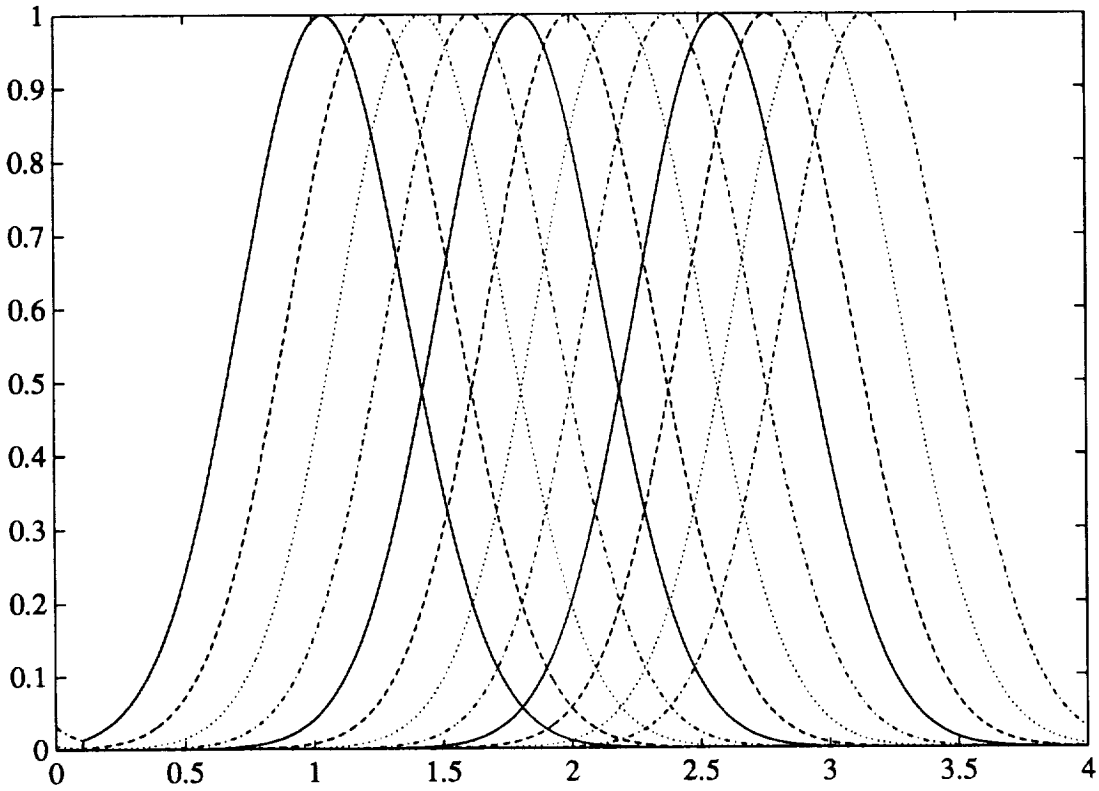

Figure 4. Example of Basis Function

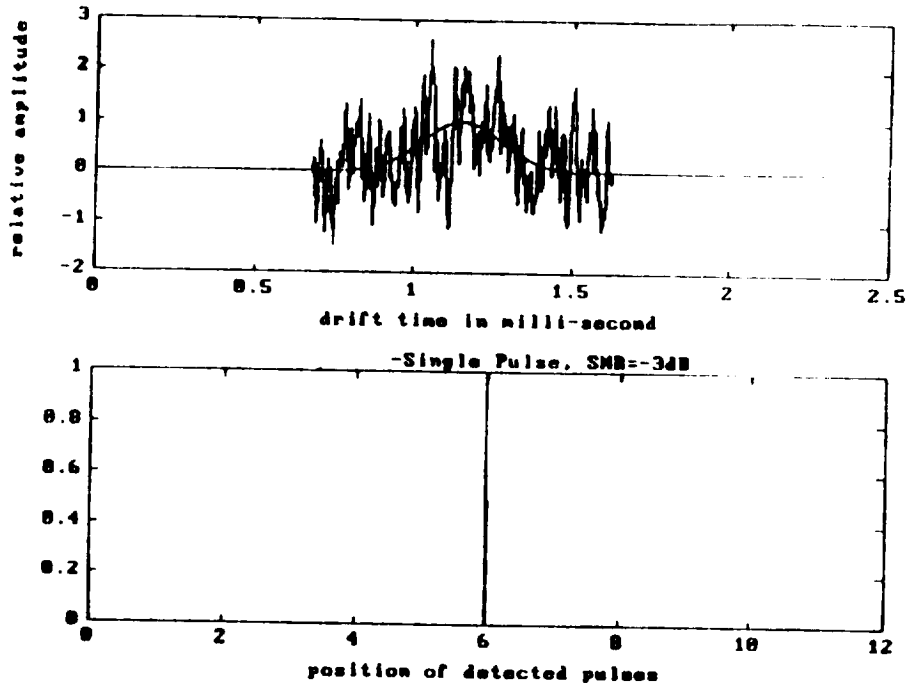

Figure 5. Hopfield Network Sensitivity to Noise: Simulated Signal

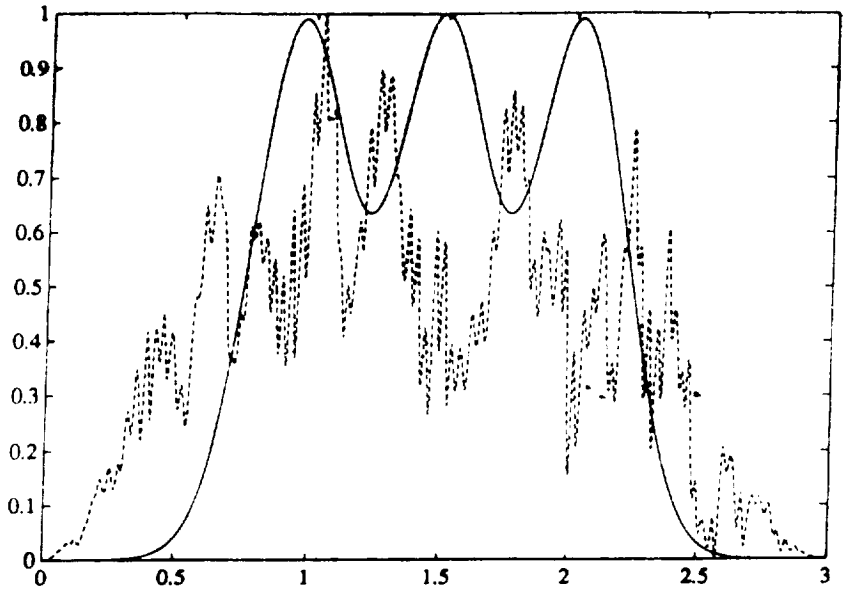

Figure 6. Hopfield Network Result on Blank Signal from Ionscan Instrument

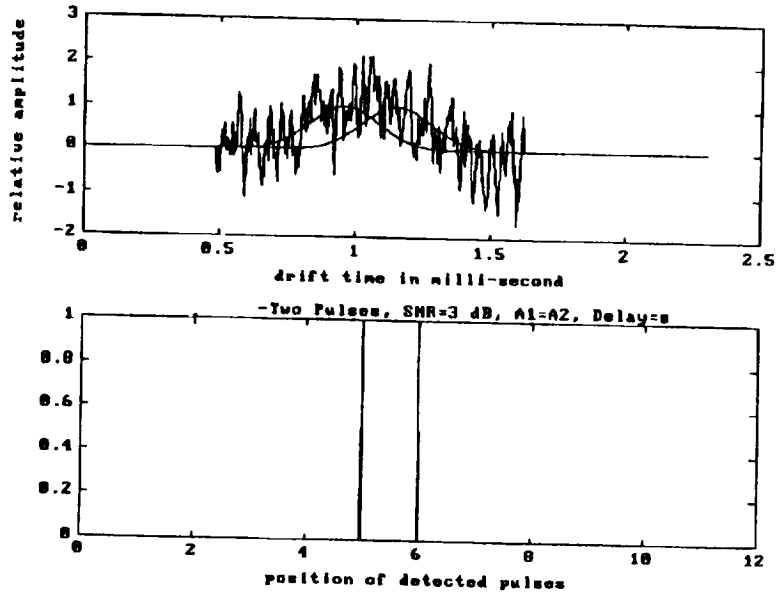

Figure 7. Resolving Power of Hopfield Network: Equal Amplitudes, SNR = 3 dB

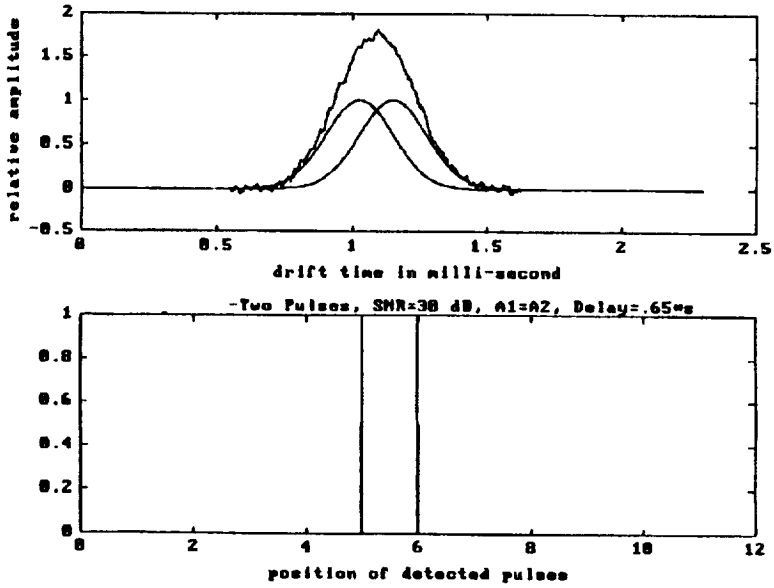

Figure 8. Resolving Power of Hopfield Network: Time Resolution of 0.65 σ

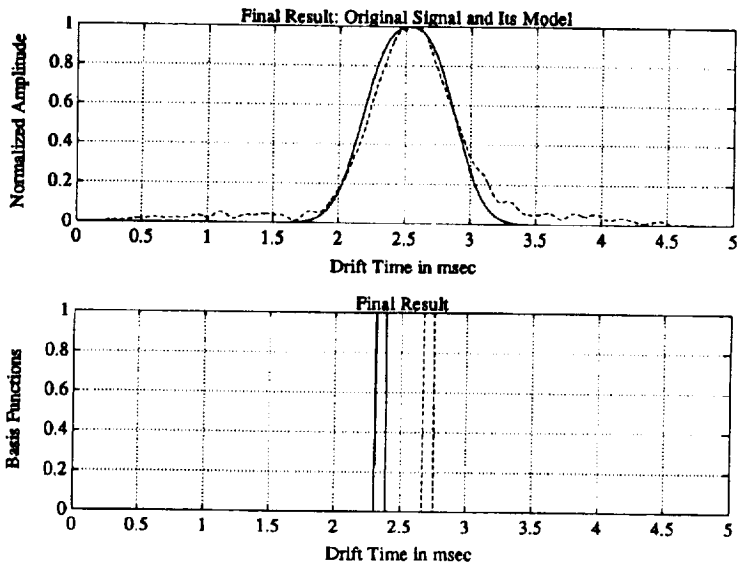

Figure 9. Resolving Power of Hopfield Network: Separation of Cocaine and Tetracaine.