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Feasibility of Using Infrared Spectroscopy and Pattern Classification for Screening Organic Pollutants in Waste Samples

FEASIBILITY OF USING INFRARED SPECTROSCOPY AND PATTERN CLASSIFICATION FOR SCREENING ORGANIC POLLUTANTS IN WASTE SAMPLES

by

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ABSTRACT

This project was undertaken to determine the feasibility of using pattern classification techniques and infrared spectroscopy to screen hazardous waste samples in the field. The technique would require a portable IR spectrometer and a microcomputer to perform a binary pattern classification of the spectra. The classification scheme requires "training" on a main frame computer to produce weighting vectors from infrared library spectra. The weighting vectors, when applied to pattern vectors obtained from sample spectra, could classify samples in the field as being likely or not likely to contain hazardous substances as defined by the spectral library.

Preliminary tests of the scheme using 50 compounds from the U.S. Environmental Protection Agency Priority Pollutant List are encouraging. The ability of the simple, linear, binary pattern classification scheme to predict whether a compound is in the class known as hazardous pollutants appears feasible.

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CONTENTS

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SECTION 1

INTRODUCTION

This feasibility study was part of a larger project jointly funded by EPA and NASA under an interagency agreement entitled Electronic Methods for In-Situ Monitoring of Hazardous Wastes. Two approaches were under investigation, x-ray fluorescence spectroscopy and infrared spectroscopy. Martin-Marietta, Denver Division, was the prime contractor (to NASA) and was responsible for both efforts. The infrared feasibility study was subcontracted to Coloraado State University and composed only 5% of the total project budget, the major effort being the development of x-ray fluorescence spectrometry as a viable field screening technique for hazardous wastes.

The goal of this project was to perform a feasibility study to determine whether it is possible to screen environmental samples, especially industrial wastes and sludges in the field, and thus to determine if hazardous pollutants are likely present. The proposed instrumental technique is infrared spectroscopy, most likely some form of Fourier transform infrared spectroscopy. The proposed decision making technique is pattern recognition or pattern classification.

SECTION 2

CONCLUSIONS

By using a limited data set of infrared spectra and limited time, it has been determined that the ability of a simple, linear, binary, pattern classification scheme to predict whether a compound is in the class known as hazardous pollutants appears feasible.

This study also has shown that preliminary investigations using infrared spectra and pattern classification schemes can be conducted on a microcomputer.

SECTION 3

INFRARED SPECTROSCOPY

The coupling of infrared and pattern classification has precedents in the literature (1,2). A brief introduction will be given for each.

INFRARED SPECTROSCOPY

Organic molecules contain a variety of forms of energy. One of these is that manifested as vibration of the chemical bonds. The absorption of electromagnetic radiation in the region known as infrared (2.5-15 micrometer wavelengths) can cause transitions in the level or state of these vibrations. Scanning through this wavelength range results in a plot of absorption versus wavelength, or an infrared spectrum, which is characteristic of the compound or mixture of compounds in a sample. Fourier transform infrared spectroscopy is an instrumental and mathematical method of collecting many such scans in a short period of time, thus improving the signal-to-noise ratio. The signal-to-noise ratio increases proportionally to the square root of the number of repetitive scans. Thus, for example, by scanning 100 times, improvement by a factor of 10 is usually realized experimentally. As a result of Fourier transform techniques, it is reasonable to expect to obtain a spectrum from less than microgram quantities of many types of organic molecules. Thus, infrared spectroscopy has found use in environmental analyses (3). It is expected that in

many types of matrices, a few hundred parts per billion of several molecular types can be detected, but not quantitatively determined. The detection limit will depend upon the type of infrared chromophore (color producing group) in the molecular structure.

PATTERN CLASSIFICATION

The availability of high-speed computers for processing large amounts of data has led to the consideration of volumes of data which were previously implausible to treat. One outcome of this ability has been the use of pattern recognition or pattern classification techniques in chemistry. According to Jurs and Isenhour (4), pattern recognition "includes the detection, perception, and recognition of regularities (invariant properties) among sets of measurements describing objects or events." Pattern recognition is normally used by chemists and others to classify a set of experimental data as a member of a class. This technique has been applied to many types of problems.

A basic pattern recognition system usually contains the units shown in Figure 1 (4, p.3).

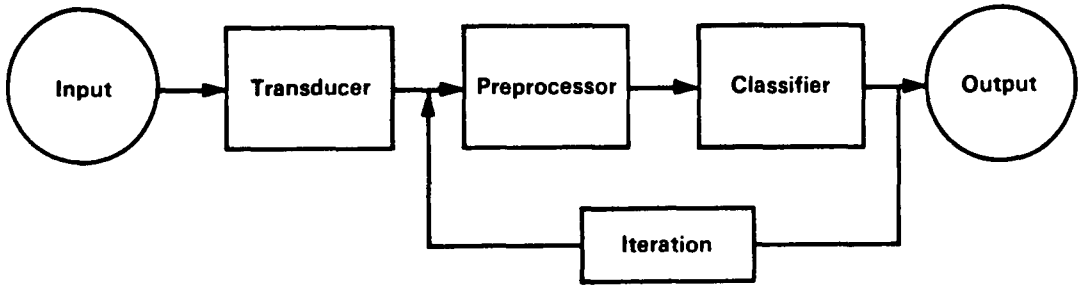

Figure 1. Block diagram of a basic pattern recognition system.

The transducer converts information from the laboratory format into the pattern space of the pattern recognition system. Often, this entails no more than converting the raw data into a suitable computer format. The preprocessor accepts the data and converts it into a form which is dealt with more easily by the classifier. The classifier treats the data by some algorithm to produce a classification decision. The classifier may be based on various branches of applied mathematics, statistical decision theory, information theory, or geometric theory. There exists a variety of pattern classification systems including those for multicategory classification. However, in this report only a binary classification system is considered and discussed.

The object of this feasibility study is to determine whether the presence of hazardous organic pollutants such as, but not limited to, those on the Environmental Protection Agency Priority Pollutant List can be predicted from an infrared spectrum of industrial waste samples. Thus, only a binary classifier is required to determine whether or not the samples contain such compounds. The hazardous pollutants often contain such organofunctional groups as C-Cl bonds, phenolic groups, polyaromatic hydrocarbons (PAH's) and other structural units represented in infrared spectra. Usually, determining even the likely presence of such compounds requires extensive preanalytical separation for successful detection by IR spectroscopy. A fast inexpensive method of sample classification could be an effective cost-saving aid.

Chemical data such as infrared spectral information may be represented as a d-dimensional pattern vector:

$$X = x_1, x_2, \ldots x_d \tag{1}$$

The components x_j are observable quantities such as the wavelength of a peak in an infrared spectrum of a compound. Alternatively, the spectral region may be divided into subregions, and the x_j values would then represent the intensity of the absorption in each subregion. If there were 100 such subregions, there would be 100 dimensions of data, or a set of vectors in 100-space, one set for each of the subregions of the infrared spectrum. If thousands of compounds are considered, clearly a vast amount of data could result.

For a binary classifier, the two classes of data should fall on either side of a decision surface. For a simple two-space case, this amounts to tracing a line (not necessarily a straight one) that runs between the two classes of data. In hyperspace, the analogy is a hyperplane that may or may not be linear and separates the two classes of data. The case is simpler if a linear hyperplane can be used as it can be represented by a vector from the origin. In such a case, the sign of the dot product of the normal vector W and a pattern vector X defines on which side of the hyperplane a given pattern point lies (4, p.11):

$$W \cdot X = |W| \; |X| \cos \theta \tag{2}$$

where θ is the angle between the two vectors. Since the normal vector is perpendicular to the hyperplane, all patterns having dot products that are positive lie on the same side of the plane as the normal vector, and all those with negative dot products lie on the opposite side. Although decision

surfaces need not be linear, their simplicity is appealing.

Often a concept called Threshold Logic Units (TLU) is used for linear, binary classification. This method uses some function which generates one of two results based on the input data. A decision is based upon whether the result is greater or less than the threshold value. The result may be computed by weighted components, w_d, of the normal vector, W, applied to each term in the data set

$$W \cdot X = |W| \ |X| \cos \theta = w_1 x_1 + w_2 x_2 + \ldots + w_d x_d + w_{d+1} \qquad (3)$$

where W_{d+1} is added to project the vector from the origin. The weight components are determined by "training" the classifier with a set of data of known classification. These data are known as the "training set" which is considered by the classifier one set at a time. The weight vector components w_d are adjusted until the prediction is correct. All pattern points in the training set are iterated until the discriminate function converges on one that successfully classifies all the points. Figure 2 shows a two-space representation of a linear, binary classifier using a TLU.

When convergence is obtained, weight vector components for each element in the data set are available. To execute the training set, it is obviously best to use a large, fast computer. However, especially for projects of the type considered here, it is important to note that once the training is complete, the weight vector components can be stored in a very small microcomputer, and prediction can be made on new data acquired using an instrument such as an FTIR spectrometer. It is conceivable that small, single-board microcomputers could

be used with a single Read Only Memory (ROM) for the program and data.

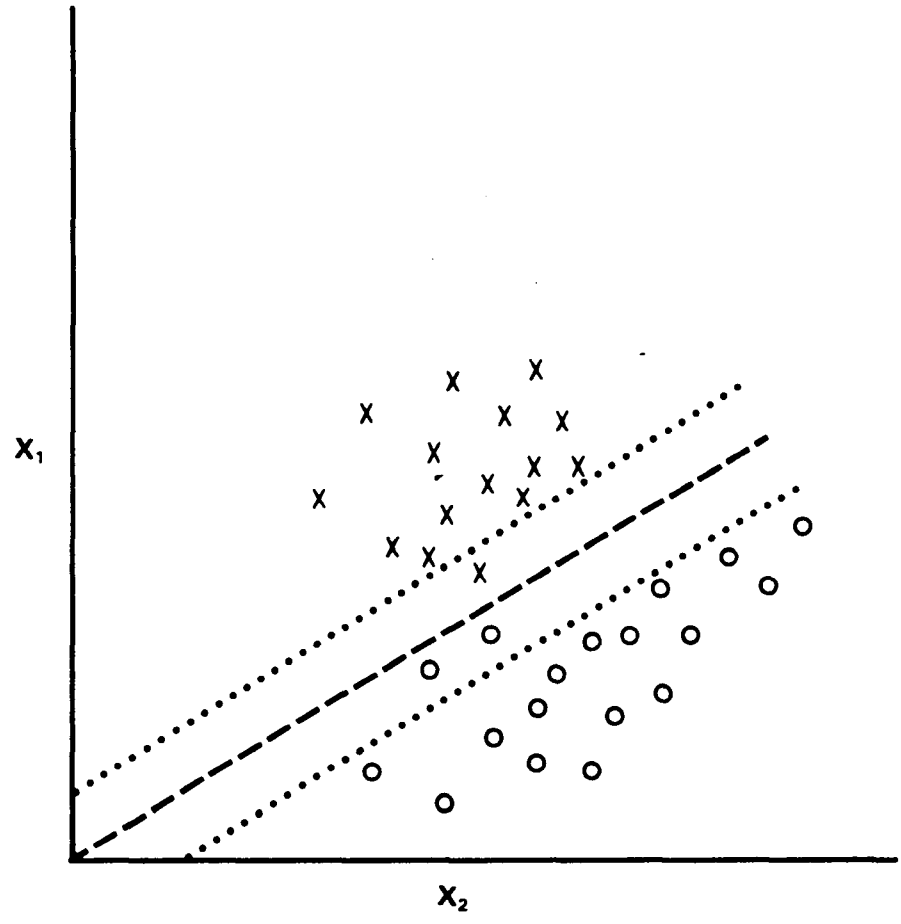

Figure 2. Example of a two-space, linear, binary classification. Two classes of data represented by x and o, respectively, fall on either side of the decision plane represented by the dashed line. An upper and lower threshold (TLU) are represented by dotted lines. Data which fall between the threshold limits are not classified.

SECTION 4

EXPERIMENTAL

This feasibility study was performed with limited resources. However, the success that was obtained illustrates the possibility of using small computers for the application of using pattern classification and infrared spectrometry to screen hazardous waste samples in the field. Appendix A shows the listing of a computer program for linear, binary pattern classification written in Apple Computer Applesoft language. This program was translated from the FORTRAN program given in the appendix of the book by Jurs and Isenhour (4). The program was executed on an APPLE II+ (Apple Computer, Cupertino, CA) computer.

The original plan for this study was to use a computer data station from a vendor of infrared instrumentation along with infrared data on diskette. Several unfortunate events occurred. The liaison from the vendor failed for several months to arrange the loan of a data station. Once obtained, no software support or manuals were available. The form of the data on the diskettes was found to be unsatisfactory for use in a classification program. Therefore, as described below, an alternative was found. Although not considered to be completely satisfactory, a meager amount of data were utilized which shed some insight to the question at hand.

The spectra for 100 compounds were encoded for use in this study. Fifty

compounds were selected from the Environmental Protection Agency Priority Pollutant List. An additional 50 compounds were selected which are not on either the EPA Priority Pollutant List or in Appendix VIII, 40CFR261 (RCRA). Data for the Priority Pollutants were derived from spectra published by Sadtler (5) and data for the other compounds were derived from spectra published by the Aldrich Chemical Co. (6). The infrared spectra of these compounds were divided into eight regions (Table 1). Each region is in units of cm^{-1}, and the data entry is a one (1) if a peak is present in the region and zero (0) if no peak is present in the region.

TABLE 1.

Spectral Region	Range of Wave Numbers (cm^{-1})	Spectral Region	Range of Wave Numbers (cm^{-1})
1	200-500	5	2001-2500
2	501-1000	6	2501-3000
3	1001-1500	7	3001-3500
4	1501-2000	8	3501-4000

The data set was assigned a "dot product" or class of one (1) if the compound were a hazardous pollutant, or a negative one (-1) if it were not. A training set was made up from 80 of the 100 compounds, 40 from the Priority Pollutant List (hazardous), and 40 from the Aldrich library (nonhazardous). This left the spectra of 20 compounds (10 from each classification) to be used as test data. Although this is a meager and greatly simplified data set, the results are encouraging. These data were analyzed using the program shown in Appendix

A, requiring approximately 45 seconds to execute on the APPLE II+ computer.

SECTION 5

RESULTS

The results of the use of the data described above executed in the Applesoft program are shown in Appendix B. The first line indicates that 60 data sets are to be used in training, that there are eight data in each set, and that the TLU has been set to 0.75 on each side of a linear surface. The nine weight vectors (including the w_{d+1} component) are the weight vectors for each datum. There were 26 feedback iterations to determine the weight vectors; each were set initially at 0.1 in line 130 of the program. With a deadzone (TLU) about the decision surface of 0.75, 13 of the 20 data sets were predicted and 7 were not. Of the 13 predicted, 1 was predicted incorrectly. With a deadzone (TLU) about the decision surface of 0, 20 of the 20 data sets were predicted and 5 predicted incorrectly. Thus, with this simple set of data, 75 percent of the test set were correctly predicted with a training of 26 feedbacks.

Appendix C shows a modified run of the program in which 70 data sets were used as the training set and a TLU of 1 was specified. The increased TLU increased the magnitude of the weight vector components which has the effect of spreading the vectors in hyperspace. All 10 compounds of the test set were predicted when 500 feedbacks were allowed, but when the TLU was reduced to zero, 3 were incorrectly predicted.

Appendix D shows the results of a run in which 80 data sets were used to train for the prediction of 20 data sets, all of which were known to fall into one of two classes. All 20 compounds of the test set were predicted correctly with 100 iterations and the TLU set at both 0.75 and zero.

Although this feasibility study was not as extensive as desired because of a variety of problems including limited funding and delays in the loan of equipment, some encouraging results were obtained. If an appropriate number of training data were to be used, the execution time on a microcomputer would be prohibitively long, but this study shows that preliminary work can be conducted on such a computer. The majority of the computer time is spent in the training session. Once the weight vectors are obtained, the prediction takes only a few seconds to determine, as this is a direct, not an iterative computation. Clearly, a small microcomputer such as those associated with modern spectrometers can perform this computation. Most importantly, although the data set used was small, the ability of the simple, linear, binary pattern classification scheme to predict whether a compound is in the class known as hazardous pollutants appears feasible.

SECTION 6

SUGGESTED FURTHER RESEARCH

The results and conclusions of this feasibility study suggest the probable success of further research. Provided that an infrared spectrometer containing even the most basic microcomputer can be designed with sufficient sensitivity and portability, a research plan to develop a system for the rapid, inexpensive, and reliable screening of hazardous waste samples for as little as a few micrograms of organic pollutant is recommended. First, a large data file of infrared spectra suitable for use in a pattern recognition scheme would be obtained on a lease basis. The most obvious of these data bases is that from Sadtler. The general pattern classification program "ARTHUR"[a]/ would be obtained for execution on a large mainframe computer. This program permits the use of a wide variety of pattern classification techniques. Therefore, one would not be restricted to the linear, binary classification used here. However, linear, binary classification would be explored in detail first because of its mathematical simplicity. The judicious use of asymmetric TLU's would be explored to "bias" the decision to predict the presence of probable pollutants even when they might not be present, if that were a desired result.

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[a]/ ARTHUR is a generalized pattern classification program available from Infometrix, Seattle, Washington. It is planned to be made available in a microcomputer version.

These studies would be required on pure compound spectra first. Then, computer-generated spectra of mixtures simulated by linear addition of the spectra of pure compounds would be investigated. For example, the reliability of the prediction when a trace of pollutant was mixed with a large amount of some other compound would be tested. This would be a severe and critical test. Fortunately, it can be performed using computer-generated data. Preparation of laboratory mixtures would only be necessary to test the instrumentation.

It is estimated that this research could be conducted during one year at a cost of approximately $70,000.

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APPENDIX A

```
20  REM  ************* BINARY PATTERN RECOGNITION ************************

30  REM
40  REM
49  REM    ***************************************************
50  REM  THIS PROGRAM WAS TRANSLATED FROM
51  REM  A FORTRAN VERSION IN THE BOOK
52  REM  "CHEMICAL APPLICATIONS OF PATTERN RECOGNITION"
53  REM  P.C. JURS AND T.L. ISENHOUR
54  REM  WILEY INTERSCIENCE, NY, 1975
55  REM  TRANSLATION BY D.E. LEYDEN
56  REM  DEPT. OF CHEMISTRY
57  REM  COLORADO STATE UNIVERSITY
58  REM  FORT COLLINS CO 80523
59  REM   ***************************************************
80  RESTORE
90  HOME
95  PRINT  CHR$ (4);"PR#1"
98  PRINT  CHR$ (9)"80N"
100  DIM D(5,100),W(6),L(100),ID(100),IC(100),NS(100),KP(20)
110 NT = 80
120 NP = 20
130 WI = .1
140 TS = .75
150 NO = NT + NP
160 NA = 1000
170 NU = 5
180  REM  READ DATA SET
190  FOR I = 1 TO NO
200  READ L(I)
210  FOR J = 1 TO NU
220  READ D(J,I)
230  NEXT J
240  NEXT I
250  REM  SET UP TRAINING SET
260  FOR I = 1 TO NT
270 ID(I) = I
280  NEXT I
290  REM  SET UP PREDICTION SET
300  FOR I = 1 TO NP
310 IC(I) = I
320  NEXT I
330  REM  INITIALIZE WEIGHT VECTOR
340  FOR J = 1 TO NU
350 W(J) = WI
360  NEXT J
370 W(NU + 1) = WI
380  REM   GOTO TRAINING SUBROUTINE
390  GOSUB 1000
400  REM  GO TO PREDICTION ROUTINE WITH DEADZONE OF .75
410  GOSUB 2000
415 TS = 0
420  REM  GO TO PREDICTION ROUTINE WITH DEADZONE OF 0.0
430  GOSUB 2000
435  PRINT  CHR$ (4);"PR#0"
440  END
```

```
1000  REM  SOUBROUTINE TRAIN
1010 NC = 0
1020  PRINT "TRAINING ";NT,NU,TS
1030 NV = NU + 1
1040 NF = 0
1050 KK = 0
1060 KV = 0
1070  REM   STARTING POINT OF MAIN LOOP & RETURN FROM LINE NUMBER 1520
1080 KZ = 0
1090  IF KV <  = 0 GOTO 1120
1100 ND = KV
1110  GOTO 1170
1120 ND = NT
1130  FOR I = 1 TO NT
1140 NS(I) = ID(I)
1150  NEXT I
1160  REM   THE NEXT LOOP CLASSIFIES THE ND MEMBERS OF THE CURRENT SUBSET
1170  FOR IR = 1 TO ND
1180 I = NS(IR)
1185  REM  THE NEXT LOOP CALCULATES THE DOT PRODUCT
1190 S = W(NV)
1200  FOR J = 1 TO NU
1210 S = S + D(J,I) * W(J)
1220  NEXT J
1230  REM  THE NEXT THREE STATEMENTS TEST FOR CORRECT ANSWER
1240  IF L(I) > 0 GOTO 1260
1250  IF (S + TS) <  = 0 GOTO 1420
1255  GOTO 1290
1260  IF (S - TS) > 0 GOTO 1420
1265  REM  1270 OR 1290 CALCULATES THE CORRECTION INCREMENT
1270 C = 2 * (TS - S)
1280  GOTO 1300
1290 C = 2 * ( - TS - S)
1300 XX = 1.0
1310  FOR J = 1 TO NU
1320 XX = XX + D(J,I) ^ 2
1330  NEXT J
1340 C = C / XX
1350  REM  THE NEXT LOOP PERFORMS THE FEEDBACK
1360  FOR J = 1 TO NU
1370 W(J) = W(J) + C * D(J,I)
1375  NEXT J
1380 W(NV) = W(NV) + C
1390 KZ = KZ + 1
1400 NS(KZ) = I
1410 NF = NF + 1
1420  NEXT IR
1430 KV = KZ
1440 KK = KK + 1
1450 KP(KK) = KV
1460  IF (KK - 20) < 0 GOTO 1500
1470  PRINT KP(KK)
1480 KK = 0
1490  REM  TEST FOR NUMBER OF FEEDBACKS
1500  IF (NF - NA) =  > 0 GOTO 1550
1510  REM  TEST WHETHER CURRENT SUBSET IS INTIRE TRAINING SET
1520  IF (ND - NT) <  > 0 GOTO 1080
1530  REM  TEST FOR ZERO ERROR
1540  IF KV <  > 0 GOTO 1080
1550 NC = 1
```

```
1560  REM  SUMMARY OUTPUT OF TRAINING ROUTINE
1570  FOR K = 1 TO KK
1580  PRINT  INT (KP(K));
1590  NEXT K
1595  PRINT
1600  PRINT "WEIGHT VECTOR"
1610  FOR J = 1 TO NV
1620  PRINT W(J)
1630  NEXT J
1640  PRINT "FEEDBACKS ";NF
1650  RETURN
2000  REM  SUBROUTINE PREDICTION
2010 L1 = 0
2020 L2 = 0
2030 KW = 0
2040 N1 = 0
2050 N2 = 0
2060  FOR II = 1 TO NP
2070 I = IC(II)
2080 S = W(NU + 1)
2090  FOR J = 1 TO NU
2100 S = S + D(J,I) * W(J)
2110  NEXT J
2120  IF ( ABS (S) - TS) =  > 0 GOTO 2150
2130 KW = KW + 1
2140  GOTO 2230
2150  IF L(I) > 0 GOTO 2200
2160 N2 = N2 + 1
2170  IF ( - S - TS) > 0 GOTO 2230
2180 L1 = L1 + 1
2190  GOTO 2230
2200 N1 = N1 + 1
2210  IF (S - TS) > 0 GOTO 2230
2220 L2 = L2 + 1
2230  NEXT II
2240  PRINT "PREDICTION WITH DEADZONE = ";TS
2250 LT = L1 + L2
2260 JW = N1 + N2
2270 PW = 100 - (100 * LT / JW)
2280 P1 = 100 - (100 * L1 / N2)
2290 P2 = 100 - (100 * L2 / N1)
2300  PRINT "NUMBER PREDICTED = ";JW
2310  PRINT "NUMBER NOT PREDICTED = ";KW
2320  PRINT "NUMBER PREDICTED INCORRECTLY = ";LT
2330  PRINT
2340  PRINT LT;"/";JW;"  "; INT (PW)
2350  PRINT L1;"/";N2;"  "; INT (P1)
2360  PRINT L2;"/";N1;"  "; INT (P2)
2365  PRINT
2370  RETURN
```

APPENDIX B

```
TRAINING 60     8                .75

WEIGHT VECTOR
-2.32670578
1.93840788
-2.80600923
-2.90653334
1.72147704
-.949600764
1.82722324
1.7296907
.512025906

FEEDBACKS 26


PREDICTION WITH DEADZONE = .75
NUMBER PREDICTED = 13
NUMBER NOT PREDICTED = 7
NUMBER PREDICTED INCORRECTLY = 1

1/13  92
1/4  75
0/9  100

PREDICTION WITH DEADZONE = 0
NUMBER PREDICTED = 20
NUMBER NOT PREDICTED = 0
NUMBER PREDICTED INCORRECTLY = 5

5/20  75
5/8  37
0/12  100
```

APPENDIX C

```
TRAINING 70      8                1
8                8
WEIGHT VECTOR
-14.1103004
50.9810323
-46.4651464
-48.268018
2.36107728
-7.01234953
5.69704767
12.8294714
-4.55528285

FEEDBACKS 500


PREDICTION WITH DEADZONE = 1
NUMBER PREDICTED = 10
NUMBER NOT PREDICTED = 0
NUMBER PREDICTED INCORRECTLY = 3

3/10  70
1/5  80
2/5  60

PREDICTION WITH DEADZONE = 0
NUMBER PREDICTED = 10
NUMBER NOT PREDICTED = 0
NUMBER PREDICTED INCORRECTLY = 3

3/10  70
1/5  80
2/5  60
```

APPENDIX D

```
TRAINING 80     5                .75
8
3
1
0322104333332222100
WEIGHT VECTOR
.605293118
.959874479
.299292939
-.713884741
-.409657796
.0221140133
FEEDBACKS 254
PREDICTION WITH DEADZONE = .75
NUMBER PREDICTED = 20
NUMBER NOT PREDICTED = 0
NUMBER PREDICTED INCORRECTLY = 0

0/20  100
0/9  100
0/11  100

PREDICTION WITH DEADZONE = 0
NUMBER PREDICTED = 20
NUMBER NOT PREDICTED = 0
NUMBER PREDICTED INCORRECTLY = 0

0/20  100
0/9  100
0/11  100
```