

AD-E950 745

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AD-A159 282



TECHNICAL REPORT RK-85-7

LIQUID CHROMATOGRAPHIC ANALYSIS OF
NITROCELLULOSE-BASE PROPELLANTS:
FINAL REPORT OF SECOND YEAR

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20000727262

23 APRIL 1985



U.S. ARMY MISSILE COMMAND

Redstone Arsenal, Alabama 35898-5000

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REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER RK-85-7	2. GOVT ACCESSION NO. AD-A159282	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) Liquid Chromatographic Analysis of Nitrocellulose-Base Propellants: Final Report of Second Year		5. TYPE OF REPORT & PERIOD COVERED Technical Report
7. AUTHOR(s) James G. Carver		6. PERFORMING ORG. REPORT NUMBER
9. PERFORMING ORGANIZATION NAME AND ADDRESS Commander U. S. Army Missile Command ATTN: AMSMI-RK Redstone Arsenal, AL 35898-5249		8. CONTRACT OR GRANT NUMBER(s)
11. CONTROLLING OFFICE NAME AND ADDRESS Commander U. S. Army Missile Command ATTN: AMSMI-RK Redstone Arsenal, AL 35898-5249		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		12. REPORT DATE 23 April 1985
		13. NUMBER OF PAGES 85
		15. SECURITY CLASS. (of this report) UNCLASSIFIED
		15a. DECLASSIFICATION DOWNGRADING SCHEDULE
16. DISTRIBUTION STATEMENT (of this Report) Approved for public release; distribution is unlimited.		
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)		
18. SUPPLEMENTARY NOTES		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number)		
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) A procedure has been developed that is able to predict the optimum conditions for separation of the ingredients of a complex mixture by liquid chromatography. The procedure involves establishing a data base of retention times for a large number of propellant ingredients in 7 predetermined combinations of 3 solvents. When a new combination of some of these ingredients is encountered, a computer analysis of the retention times of the ingredients from the data base will predict the best solvent composition to separate all the ingredients. The details of the procedure and several examples of its use are discussed.		

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I. INTRODUCTION

Nitrocellulose-based propellants are widely used throughout the military and civilian areas in guns and rockets. There are almost as many different procedures for analysis as there are propellants. Even in the area of liquid chromatography, frequently, more than one procedure is used in the analysis of the propellant. Most techniques have been designed to answer a specific question about the purity or amount of two or three ingredients. However, most propellants have at least three or four original ingredients and frequently one or two other reaction products that can be easily extracted.

Since these ingredients can be extracted into a solvent it would seem logical to try to analyse for all of them at one time. Early efforts centered on developing separations using silica columns such as Corasil II and eluting with non polar solvents such as hexane, methylene chloride, and dioxane [1-14]. A major difficulty to this in the past has been that silica columns are difficult to work with. Precise control of water, alcohols, and other polar ingredients is necessary to obtain reproducible retention times and amounts.

II. BACKGROUND

Chromatographic efforts shifted to reverse phase separations with the advent of permanently bonded phases and improved equipment. Reverse phase columns do not have the problems that silica columns do. Materials are separated on differences in their nonpolar segments rather than in their polar segments as in silica columns. Currently over 80% of all HPLC separations are performed on reverse phase columns [15], but investigators have expressed difficulty in obtaining good separations of complex mixtures with the binary solvent mixtures commonly used [16-18]. Nonetheless, analysis times have been shortened from 10 to 12 hours using the older MIL-STD-286B methods to less than one hour using HPLC [19-20].

Recently, significant interest has been observed in using more than 2 solvent systems with C-18 columns. Manufacturers have started producing HPLC equipment that can pump 3 or 4 solvents simultaneously. The reason for a multi-solvent system is that separations are accomplished by more than differences in polarity. Snyder [21] has proposed that solvents should be classified as to three properties: proton acceptor, proton donor, and dipole interaction. It is believed that these properties make the major contributions to separations in HPLC. Kirkland [22] has developed a procedure consisting of 7 experiments which can be used to predict the best solvent composition for separating ingredients in a mixture. A program was undertaken to develop a procedure that will separate the maximum number of commonly used propellant ingredients possible.

Previously [23] a procedure was described where with a series of 7 experiments the separation of up to 10 propellant ingredients would be optimized. The procedure is not one that should be used for every unique analytical problem encountered but shows great promise for the occasionally difficult cases and for repetitive analysis. In production facilities this latter case will be of significant aid. Frequently, several samples of the same material require rapid analysis each day. It is not unusual for more than one formulation to be in production at the same facility. Isocratic separations are desired for their reproducibility and turn around time, also no column

reequilibration is required. If one set of conditions could be used for all the formulations manufactured at the facility, the samples could be analysed in a random or as received order rather than in groups.

At the time of writing the first paper on this subject several limitations still existed. The computer program employed could only work with 10 ingredients and was limited to a 64K mini computer operating in Basic. This limitation was imposed by computer memory restrictions. A disc based computer was recently made available to the RD&E Center laboratory. With the computers increased capabilities for large matrix operations it was possible to modify the program to consider many more materials simultaneously. A limit of 35 ingredients was arbitrarily selected as the maximum number for calculations. A program this size performs a complete evaluation to 595 pairs of ingredients. As a practical limit 20 propellant ingredients were selected for further work.

A new disc based program has now been written. It includes several improvements over the original program such as calculating with the actual peak widths rather than an average, and plotting all resolution lines on one graph on a x-y plotter. This program has been written in Basic. A Fortran 77 version is also available. See Appendix B for Basic programs "XLDB6 and XLDB6B", see Appendix C for Fortran 77 program "LC Main".

III. EXPERIMENTAL

The chromatographic system consisted of a Waters Associates 720 System Controller, 710B WISP sample injector, three M6000 pumps, a Perkin-Elmer LC 85 variable wavelength ultra violet detector and either a Supleco 250 mm x 4.6 mm ID Supelcosil 5 micron C-18 column or a Dupont 5 micron ZORBAX ODS 4.6 mm x 250 mm column. Solvents were from Burdick and Jackson and were filtered prior to mixing. Solvents in this study were combinations of the identified solvent and distilled deionized water. MeOH is 60% methanol-40% water, ACN is 50% acetonitrile-50% water, and THF is 42% tetrahydrofuran-50% water.

The analog UV signal was digitized and sent to a Hewlett-Packard 3357 Laboratory Automation System for data reduction. Following normal integration, a computer program was automatically run that calculated the peak widths and retention times required for input to the solvent selection program (see Basic program "Peak WT" in Appendix A).

Samples were prepared by cutting a propellant into small pieces and accurately weighing 50-100 µg into a 15 ml test tube. An accurately weighed internal standard was added to the test tube and then 10 ml of a 50% acetonitrile/water solution was added. An ultrasonic micro tip was placed in the test tube and the sample was extracted for 15 minutes. The solution was filtered and placed in a WISP sample vile for analysis.

A 50% acetonitrile solution was selected because noncross linked nitrocellulose samples will dissolve completely or form a gum in pure acetonitrile. If this solution is filtered or injected onto a column the nitrocellulose would plug the pores.

When HMX is suspected in the sample and quantitative analysis is required, a smaller sample or greater amount of solvent is needed. HMX has only a limited solubility in even pure acetonitrile. RDX is much more soluble than HMX and should not present a problem. You must allow for the presence of HMX and RDX even when no qualitative data is required, when selecting the optimum chromatographic solvent.

IV. SOLVENT SELECTION

A column is first characterized by determining the retention times and peak widths of all known propellant ingredients in 7 predetermined solvent compositions. This data is fed into a computer file for later reference. This only needs to be done once for each column (see Table 1). This procedure has been described earlier [22].

Most samples are submitted for analysis in RD&E Center laboratory as part of an aging surveillance program or for verification of composition. In these cases the computer is asked to analyse the data file for only those ingredients known or suspected to be present in the propellant extract. A typical example of this procedure is the N-5 propellant.

The first propellant is known to contain Nitroglycerine, Diethylphthalate, and 2-Nitrodiphenylamine. The binder, lead salts, and other insoluble ingredients are not considered. As the propellant ages, degradation products of 2-NDPA are expected to be produced. Therefore, the computer was asked to predict the best conditions for separation of 2,2-Dinitro DPA, 2,4-Dinitro DPA, and 2,4,4-Trinitro DPA, an internal standard Dipropylphthalate, as well as the three original ingredients. It predicted the best solvent composition for the separation would be 82% MeOH/18% ACN with a minimum resolution of 2.855 for any two peaks (see Table 2). The triangular graph (Figure 1) indicated good resolution could be obtained with MEOH/ACN combinations between 86/14 and 78/22. Since methanol has a higher viscosity than acetonitrile the 78/22 mixture was selected. This permitted a higher flow rate than possible with the 82/18 mixture.

As can be seen in the chromatograph of the standard sample (Figure 2), all the ingredients were well resolved with the 78/22 mixture at 2.0 ml/min. Once the system was calibrated the propellant samples were analysed.

V. DISCUSSION

One aspect of the procedure that has been receiving attention is its ability to predict optimized conditions for any combination of ingredients in a data base. Once the retention times of a large number of ingredients have been determined for the 7 solvent compositions, the optimum conditions for any combination of those ingredients can quickly be predicted without further experimentation. A drawback to this is the data base for a Waters 10 micrometer C-18 radial pack is not necessarily comparable to another column such as a Waters 10 micrometer steel column. To explore how significant column differences effected the predicted optimum conditions a data base was established for several different columns. Tables 3 thru 7 list the retention times for 20 propellant ingredients in the 7 solvent compositions. As reported earlier, MeOH signifies 60% methanol in water, ACN signifies 50% acetonitrile in water, and THF signifies 42% tetrahydrofuran in water. Table 8 lists the important

specifications for the 5 columns tested. Three Waters 10 micrometer C-18 Radial Pac columns were tested. The computer predicted almost exactly the same optimum conditions for all three. The obvious implication is that for these bulk loaded columns the column to column reproducibility is very good. It should be noted however that this data was obtained using a RCM-100 modular. The Z-modular uses slightly different columns and produce different results even though they have the same name.

Following the procedure described earlier, the computer predicted optimum solvent compositions were determined for each of the 5 different columns. As seen in Figure 3 most of the columns have a optimum composition in the same general area.

Not unexpectedly, a significant improvement in resolution was obtained in changing from a 10 micrometer packing to a 5 micrometer packing. Figure 4 is a chromatographic run using a 5 micrometer Waters C-18 Radial Pac column at the optimum solvent composition. Figure 5 is a run of the same solvent but on a 10 micrometer C-18 Radial Pac column.

Eighteen ingredients can be separated well enough to quantify at the predicted composition for the 5 micrometer Waters column (Figure 4). Two pairs of these peaks appear as fused peaks while the remaining ingredients are well resolved. Only ethylcentralite and diphenylamine are unresolved at this composition. In Figure 5, the 10 micrometer Waters column shows almost no completely resolved peaks for the same conditions.

At the predicted optimized conditions for the Perkin-Elmer HS-5 C-18 column we see that several of the later peaks have shifted (Figure 6). Nitroglycerine has fused with 2,4 DNT and ethylcentralite with TNT. Diphenylamine has eluted later and is almost resolved completely from BTTN. Four pairs are completely unresolved: resorcinol and triacetin; RDX and methylcentralite; 2,4 DNT and nitroglycerine; TNT and ethylcentralite. Generally the ingredients appear to be less resolved than in the Waters 5 micrometer radial pac system.

A suplecasil LC-18 column (Figure 7) was optimized for separating the mixture. Again the predicted solvent composition was similar to the previous examples. Comparison with the Perkin-Elmer column shows that ethylcentralite and TMETN have shifted relative to the other ingredients. Diphenylamine is completely resolved as is TNT but now the ethylcentralite is co-eluting with BTTN. The resolution of fused peaks is better than in the Perkin-Elmer column but there are still 4 pairs of co-eluting peaks.

The Dupont Zorbax ODS column had a predicted optimum significantly different from the others. Figure 8 indicates complete resolution of all the ingredients. Dupont literature discusses the unique properties of their Zorbax columns. They are manufactured with monofunctional silane units to produce a "monolayer". Whether or not the characteristics of this column are unique or common to all Zorbax ODS columns will have to be determined. The long elution times of the last 4 ingredients is undesirable but if the analyst wishes to use an isocratic system this situation is unavoidable. A change in the solvent composition after 17 minutes could cause these ingredients to elute faster with minimal loss of resolution. This would introduce an equilibration delay between runs however, so a trade off would have to be

made. Using the solvent composition chosen for the Supelco column, this Dupont column produced a chromatogram similar to Figure 7 except that nitroglycerine and ethylcentralite appear as fused peaks to 2,4 DNT and BTTN respectively.

Figure 9 represents a chromatogram of the Waters 10 micrometer Radial Pac column at its best composition near the selected Dupont composition. There is some shifting of peaks but the chromatograms appear similar. The most important difference is the significant loss of resolution. There are 4 fused peak pairs and 4 sets of unresolved peaks. While on first glance Figure 9 appears to be better than Figure 4, close examination indicates more partially resolved ingredients than in Figure 9. So the original predicted optimum composition for the Waters column is indeed better than the alternate.

VI. ANALYSIS OF UNKNOWN

Occasionally, a propellant of unknown composition is submitted for analysis. A variety of analytical tools are employed for this task.

For LC analysis, the solvent capable of separating, at least partially, all known ingredients is initially selected. Retention times of the unknown peaks are compared with the predicted retention times. The retention times of the authentic ingredients are then obtained for comparison. Once a list of probable ingredients is obtained, the computer selects a second solvent to separate these specific materials. Standards are then prepared and the analysis proceeds normally.

One foreign propellant (#1) analysed in this laboratory was found to be a rather simple double-base composition. The extractables were identified as nitroglycerine, NDPA, and methylcentralite. Excellent resolution was easily obtained with 100% MeOH.

A second foreign propellant (#2) provided a greater challenge. After some work it was found to contain DEGDN, methylcentralite, 2,6-DNT, 2,4-DNT, 2,4,6-TNT, nitroglycerine, NDPA, and DPA. An internal standard of Dipropylphthalate was selected. A solvent composition of 60% MeOH-40% THF was chosen. Figure 10 shows the resulting chromatogram.

A third foreign propellant (#3) turned out to be a single base nitro-cellulose propellant stabilized with diphenylamine (Figure 11). The interesting point is that the propellant appears to be contaminated with trace amounts of HMX, RDX, MNA, TMETN, 4-NDPA, and 2-NDPA. NDPA was also found but it is probably a degradation product from the DPA. Blanks were prepared and the contamination was only found in the propellant extract. It was concluded that the manufacturing facility was also engaged in production of other more sophisticated propellants.

VII. COMPARISON OF TECHNIQUES

Since the analysis of propellants has a greater confidence level when similar values are obtained by two different procedures, GC analyses of several propellants were obtained for comparison with the LC data.

N-5 samples aged in tropic, arctic, or desert conditions were examined by capillary GC for 2-NDPA to determine their remaining shelf life. LC analysis of the samples included nitroglycerine, diethylphthalate, 2,2-Dinitro DPA, and 2,4-Dinitro DPA as well as 2-NDPA. The results indicate good agreement for the two techniques (Table 9). As seen in the table and chromatograms (Figures 12, and 13), formation of the 2-NDPA degradation products proceeded further in the hotter temperature conditions and agrees with the decrease of 2-NDPA. A potential method for an internal check is to compare the total amount of 2-NDPA and its products with the initial amount of 2-NDPA in the propellant. The millimolar ratios of 2,4-Dinitro DPA and 2,2-Dinitro DPA are identical at 2.62 for the desert and tropic samples. The millimolar amounts of 2-NDPA and its products are the same for the arctic and tropic at 8.37 and 8.43, respectively. The desert is out of line with 9.29 millimoles per 100 μ g of propellant. A time zero analysis for this lot is not available; therefore, it is difficult to determine whether the discrepancy is due to a greater amount in the initial propellant or an error in the analysis. As pointed out above, the molar ratios of dinitro products are the same for desert or tropic and the total amount of 2-NDPA and dinitro products is the same for arctic or tropic. Therefore, the most likely explanation is a different amount of initial 2-NDPA.

CHAPARRAL propellant has been examined by both GC and LC techniques in RD&E Center laboratory. Several aged motors of different production lots were analysed to determine residual levels of MNA, 2-NDPA, and 4-NDPA. Additionally, the levels of nitroglycerine, BTIN and nitroso MNA were determined. Table 10 presents a summary of the results. Generally, the two procedures agree within a few percent of each other for MNA and n-MNA, but there are a few cases where as much as 9% disagreement was observed. The cause of this is not immediately clear. The total molar amounts of MNA and n-MNA as determined by the two techniques do not show a pattern other than that when aged hot the LC sees less material in the propellant than the GC and when aged at ambient the LC see more material than the GC. This may be caused by the incomplete separation of MNA and HMX. Further study will be needed to answer this question.

The SPRINT propellant is a composite modified double-base propellant. Analysis was performed for nitroglycerine, 2-NDPA, triacetin, resorcinol, and 2-nitroresorcinol. The latter was not separated by GC and the different response factors may be responsible for the different values in LC and GC. The triacetin does not absorb strongly in the UV and while resolved fairly well small fluctuations in the base line or near by peaks could significantly alter the calculated value (Figure 14). Generally, however, there is good agreement between the two techniques (Table 11).

Two foreign propellants were analysed by HPLC and capillary GC. The analyses are given in Tables 12 and 13. As mentioned earlier, GC is not able to resolve some nitroderivatives from their parent compounds where LC can. Here NDPA and DPA present such a situation. In Foreign Propellant #1, GC found 0.15% DPA while LC found no DPA and 0.40% nitroso DPA. In Foreign Propellant #2, GC found 0.47% DPA while LC found 0.36% DPA and 0.25% NDPA which corresponds, after correcting for molecular weight differences, to 0.57% DPA in the initial propellant. Again, there is otherwise generally good agreement between the LC and GC techniques.

M-36 propellant points out one of the important limitations of LC. Even if it can be separated, measurement is impossible if it is not visible. Currently, liquid chromatography has only a few detection methods available. UV is the most popular, but many compounds of interest do not absorb well so they are detected poorly. Dipropyladipate is one such compound and as discussed above triacetin is another. The chromatogram in Figure 15 indicates that dipropyladipate can indeed be well resolved from the other ingredients in M-36 propellant. Table 14 indicates that while several GC techniques using flame ionization detectors obtained an average value of 3.02% di-n-propyladipate with 0.5% relative standard deviation, LC came up with 4.04%. This is more than a 33% difference between techniques. It is hoped that newer LC detectors such as electrochemical or micro LC/Flame detectors can solve this problem.

VIII. UNIVERSAL PROCEDURE

No truly universal procedure exists. In a production facility such as Radford AAP several different formulations are being manufactured simultaneously. These formulations are chemically different only in the amount of a limited number of ingredients. Typically several solvent compositions would be required for HPLC analysis of these formulations. It is very probable that with the above described procedure one solvent composition could be quickly selected that would be able to separate all the materials in all the formulations. Thus, samples could be analysed as received or in a random order rather than holding a sample until a group using the same solvent is assembled. As an example of this, a solvent was selected that could separate 20 of the most commonly used propellant ingredients (see Figure 7). It is clear that only one set of conditions is needed to quantify any combination of these materials.

IX. CONCLUSIONS

The procedure described here seems to have almost universal application in liquid chromatography. Once the retention times for a list of ingredients is developed no further experimentation is required. When a new propellant composition is encountered, the solvent composition necessary for optimum separation of the ingredients can be quickly predicted with the aid of a computer.

The best separation achieved to date was with a 5 micrometer spherical particle coated with a "monolayer" of C-18. Other columns of this type need to be evaluated to determine column to column reproducibility. The computer program can evaluate up to 35 double base ingredients but with the columns evaluated to date a practical limit is 20 ingredients per sample. This may be improved by using 3 micrometer columns. Employing a second selective detector such as a fluorescence or an electrochemical detector should offer the better solution however.

In a production situation this procedure can quickly predict a single solvent composition that can separate up to 20 ingredients. This should permit the analysis of all the different formulations manufactured at a facility with one set of conditions resulting in a more complete analysis and more rapid turn around time in situations presently requiring several sets of conditions or gradient elutions.

It has been demonstrated that when a new formulation of known composition is encountered, an optimum solvent composition can be predicted without experimentation once a column has been characterized. It is recommended that when a column is replaced with a similar column from the same manufacturer, the column should be tested at the optimum solvent composition for the first column. If no significant differences are observed then the same data base can be used as before. Columns from different manufacturers will probably have different characteristics and should be completely characterized routinely.

Clearly, a procedure exists that can quickly obtain good separations of the extractable ingredients from a double-base or composite modified double-base propellant. LC is capable of resolving some materials and their degradation products that GC can not. Comparison of GC and LC analyses of several propellants indicates that the two techniques give approximately the same results for those ingredients that they can both detect. LC is superior to GC in its ability to separate certain materials and to detect many nonvolatile materials, such as HMX. GC is superior to LC for detection of some materials. LC and GC techniques thus appear to complement each others weaknesses and strengthen the validity of the propellant analysis. Therefore, it is recommended both techniques be used for critical determinations. Otherwise, the analyst should determine what he wants from the analysis and select the appropriate technique.

Table 1. HPLC Optimization.

INPUT DATA
 COLUMN : SUPLECO 5u C-18 SAMPLE : GENERAL
 DATA IS CORRECTED TO 1.00 ml/min
 PEAK WIDTHS IN SECONDS IN PARENTHESES

	MEOH 1	ACN 2	THF 3	ACN& MEOH 4	THF& MEOH 5	ACN& THF 6	MEOH& ACN&THF 7
Solvent Front	1.78	1.79	1.79	1.78	1.79	1.79	1.79
1 Resorcinol	3.17 (14)	3.08 (12)	3.88 (12)	3.24 (12)	3.51 (12)	3.45 (12)	3.41 (12)
2 HMX	3.17 (14)	5.14 (17)	5.61 (14)	4.25 (13)	5.76 (14)	4.68 (13)	4.72 (13)
3 RDX	4.07 (25)	5.14 (23)	6.19 (17)	4.95 (13)	7.28 (17)	5.19 (14)	5.63 (14)
4 DEGDN	4.64 (18)	6.52 (33)	7.81 (18)	5.93 (14)	7.63 (18)	6.16 (15)	6.26 (15)
5 MNA	5.14 (19)	5.82 (18)	5.83 (15)	5.95 (15)	6.26 (15)	5.46 (14)	5.75 (14)
6 Nitroglycerine	6.49 (12)	9.08 (26)	16.03 (44)	8.50 (20)	16.07 (44)	10.15 (22)	10.73 (22)
7 Acaradite I	5.96 (24)	4.49 (21)	3.65 (12)	6.02 (15)	4.25 (14)	3.90 (12)	14.53 (44)
8 2,4,6 TNT	6.91 (24)	9.25 (26)	24.06 (64)	9.18 (20)	15.59 (44)	10.80 (28)	11.24 (39)
9 BTTN	6.95 (25)	10.21 (28)	16.17 (44)	9.66 (22)	17.23 (50)	10.69 (28)	11.97 (39)
10 2,6 DNT	7.65 (35)	8.70 (23)	11.63 (39)	9.51 (21)	11.83 (39)	8.46 (20)	9.47 (21)
11 Acaradite II	6.86 (27)	5.21 (20)	4.00 (13)	7.21 (18)	5.65 (14)	4.43 (14)	5.07 (14)
12 TMTN	9.28 (46)	12.81 (34)	25.44 (65)	12.82 (38)	24.49 (64)	14.05 (39)	16.01 (40)
13 NDPA	19.96 (49)	18.61 (40)	17.42 (44)	24.62 (64)	26.50 (64)	16.95 (40)	23.04 (58)
14 4 NDPA	17.86 (44)	15.07 (40)	14.84 (40)	21.09 (53)	22.06 (55)	13.72 (39)	18.81 (50)
15 Meth Centralite	14.64 (52)	9.39 (36)	6.51 (17)	15.32 (40)	8.04 (20)	7.01 (17)	8.69 (22)
16 DPA	36.63 (55)	19.36 (44)	11.13 (39)	38.97 (60)	16.29 (38)	12.35 (35)	18.17 (40)
17 2 NDPA	35.06 (104)	24.40 (64)	18.75 (40)	37.27 (60)	30.74 (55)	19.32 (52)	26.91 (68)
18 Ethyl Centralite	14.89 (117)	15.54 (79)	6.48 (15)	19.39 (55)	14.59 (40)	11.55 (39)	14.20 (40)
19 Triacetin	3.88 (19)	4.17 (27)	3.61 (49)	4.54 (15)	4.05 (40)	3.73 (29)	3.90 (33)
20 Tetryl	4.28 (21)	5.24 (22)	6.13 (26)	5.29 (83)	7.99 (84)	5.24 (23)	5.95 (26)
21 TEGDN	5.02 (31)	7.14 (21)	7.44 (23)	6.62 (29)	7.64 (25)	5.99 (19)	6.40 (21)
22 Nitroso-MNA	6.11 (27)	7.23 (20)	8.31 (27)	7.62 (21)	8.64 (26)	6.59 (20)	7.26 (23)
23 2 N-Resorcinol	3.83 (21)	4.20 (14)	4.89 (16)	4.35 (15)	5.17 (19)	4.45 (15)	4.63 (16)
24 2,4 DN Resorcin	1.89 (16)	1.84 (16)	1.84 (20)	2.00 (15)	2.19 (24)	1.89 (16)	2.03 (20)
25 2,4 DNT	8.07 (33)	8.68 (21)	11.61 (32)	9.68 (26)	13.20 (36)	8.57 (24)	10.04 (28)
26 2,4,4 TN DPA	13.78 (56)	15.39 (42)	10.02 (80)	20.82 (60)	29.28 (75)	16.43 (54)	20.13 (62)
27 2,4 DN DPA	17.21 (55)	16.53 (44)	19.20 (51)	22.75 (65)	23.58 (70)	14.92 (68)	19.14 (92)
28 2,2 DN DPA	21.60 (83)	18.03 (27)	12.66 (37)	26.46 (73)	24.19 (71)	14.08 (41)	19.82 (57)
29 Methylphthalate	5.44 (25)	5.85 (17)	4.62 (23)	6.64 (21)	5.33 (19)	4.84 (24)	5.22 (19)
30 Ethyl Phthalate	9.72 (42)	9.82 (27)	6.85 (22)	12.41 (37)	8.21 (94)	7.29 (23)	8.35 (115)
31 Propylphthalate	24.38 (93)	21.52 (58)	12.36 (36)	32.20 (91)	17.49 (56)	13.82 (41)	18.07 (53)

Table 2. HPLC Optimization.

9:00 AM TUE., 14 AUGUST 1984

DATA ANALYSIS

COLUMN: SUPELCO 5u C-18 SAMPLE: N-5 PROPELLANT

THE BEST COMPOSITION IS:

MEOH	ACN	THF
82%	18%	0%

THE WORST RESOLUTION AT THIS POINT IS 2.855

PREDICTED ORDER OF ELUTION

INGREDIENT	RELATIVE RETENTION TIME	
	1.00 ml/min	1.50 ml/min
Nitroglycerine	7.38	4.92
Ethyl Phthalate	11.30	7.54
2,4,4 TN DPA	17.76	11.84
2,4 DN DPA	20.57	13.71
2,2 DN DPA	24.89	16.59
Propylphthalate	29.34	19.56
2 NDPA	37.61	25.07

Table 3. Optimization of 5 Micron Waters
C-18 Radial PAC.

	MEOH	ACN	THF	MEOH/ACN	MEOH/THF	ACN/THF	MEOH/ACN/THF
TRIACETIN	1.60	3.21	6.80	1.82	7.03	1.89	4.89
RESORCINOL	1.81	3.66	6.90	1.91	7.34	2.10	5.05
HMX	1.95	3.32	3.77	2.62	4.34	3.17	3.34
RDX	2.86	3.41	4.33	3.31	6.13	3.70	4.25
DEGDH	3.40	4.51	5.96	4.13	6.19	4.56	4.77
MHA	4.12	4.23	4.30	4.51	5.39	4.15	4.66
NG	4.35	6.36	14.45	5.69	12.66	6.12	9.05
ACARADITE I	4.74	3.20	2.23	4.40	3.15	2.70	3.27
TNT	4.85	6.42	21.72	6.16	13.30	6.78	9.00
BTTH	4.88	7.12	13.40	6.65	15.66	8.53	9.77
2,6 DNT	5.99	6.26	9.70	6.58	9.96	7.20	8.28
ACARADITE II	6.19	4.11	2.66	5.55	3.62	3.21	3.85
TMETH	6.45	9.00	22.05	8.88	22.09	11.41	13.28
2,4 DNT	6.93	6.36	9.65	7.03	10.50	7.12	8.41
EC	12.15	11.21	10.10	13.30	12.98	10.51	13.45
MC	13.14	8.09	5.03	12.02	6.60	5.63	7.87
4 NDPA	13.70	11.22	12.45	15.81	23.00	11.88	17.28
NDPA	13.96	12.98	15.35	15.78	23.50	15.50	21.80
2 NDPA	29.60	17.81	17.30	26.70	28.45	17.52	27.15
DPA	31.30	16.40	10.15	27.88	14.85	11.40	17.98

Table 4. Optimization of 10 Micron Waters
C-18 Radial PAC.

	MEOH	ACN	THF	MEOH/ACN	MEOH/THF	ACN/THF	MEOH/ACN/THF
TRIACETIN	.93	1.16	1.59	1.38	1.00	1.38	1.50
RESORCINOL	.86	.90	1.05	.90	1.03	.93	.93
HMX	.93	1.40	1.57	1.40	1.67	1.37	1.51
RDX	1.40	1.53	1.82	1.62	2.49	1.57	1.66
DEGDH	1.75	2.01	2.52	2.13	2.71	1.99	2.30
MHA	2.10	1.83	1.82	2.34	2.30	1.80	2.10
NG	2.27	2.86	1.17	3.06	5.92	3.38	3.95
ACARADITE I	2.48	1.45	1.01	2.30	1.37	1.16	1.45
TNT	2.54	2.94	9.05	3.38	6.06	3.76	4.06
BTTH	2.57	3.20	5.11	3.71	6.61	3.68	4.43
2,6 DNT	3.17	2.80	4.09	3.53	4.46	2.91	3.49
ACARADITE II	3.36	1.89	1.15	3.02	1.57	1.37	1.71
TMETH	3.54	4.06	17.58	5.02	9.86	5.00	6.18
2,4 DNT	6.93	6.36	9.65	7.03	10.50	7.12	8.41
EC	12.56	7.65	4.08	16.51	6.51	4.50	7.15
MC	7.85	3.68	2.11	6.66	2.98	2.35	3.24
4 NDPA	7.59	5.12	5.74	8.97	9.17	5.17	7.48
NDPA	7.03	5.13	6.41	7.68	5.78	4.17	5.54
2 NDPA	18.17	8.24	7.11	15.37	12.91	7.18	11.22
DPA	7.88	5.93	6.41	9.08	10.34	6.14	8.75

Table 5. Optimization of 5 Micron Perkin-Elmer
HS-5 C-18 Column.

	MEOH	ACN	THF	MEOH/ACN	MEOH/THF	ACN/THF	MEOH/ACN/THF
TRIACETIN	2.27	2.31	2.96	2.43	2.99	2.75	2.68
RESORCINOL	2.27	2.31	2.96	2.43	2.99	2.75	2.68
MMX	2.54	4.21	5.63	3.58	5.22	4.28	4.09
RDX	3.37	4.51	6.34	4.15	6.75	4.78	4.84
DEGDN	3.85	5.47	8.84	4.89	6.84	5.63	5.42
MHA	4.53	5.87	5.84	5.14	5.91	4.96	5.09
HC	5.14	8.18	17.08	6.91	14.51	9.92	9.78
ACARADITE I	4.94	3.48	3.13	4.69	3.72	3.34	3.69
TNT	5.42	8.27	29.08	7.24	14.44	10.42	9.43
BTTH	5.56	9.22	18.75	8.33	16.83	10.43	10.80
2,6 DNT	6.27	7.69	12.26	7.65	10.69	8.03	8.36
ACARADITE II	6.12	4.38	3.57	5.79	4.19	3.92	4.34
TMETH	7.46	11.02	30.08	10.77	23.21	13.64	14.40
2,4 DNT	6.58	7.63	11.88	7.51	10.79	8.11	8.29
EC	12.78	12.94	11.86	14.83	13.59	11.14	12.56
MC	13.31	8.18	6.40	11.93	7.69	6.63	7.89
4 NDPA	14.55	12.75	17.34	17.06	22.20	13.33	16.65
NDPA	14.94	14.93	19.26	17.56	38.23	15.91	19.35
2 NDPA	27.86	18.96	20.99	26.54	28.22	18.05	23.17
DPA	34.23	15.75	11.86	29.06	16.02	11.96	16.30

Table 6. Optimization of 5 Micron Supelcosil
LC-18 Column.

	MEOH	ACN	THF	MEOH/ACN	MEOH/THF	ACN/THF	MEOH/ACN/THF
TRIACETIN	3.17	3.08	3.88	3.24	3.51	3.45	3.41
RESORCINOL	3.17	3.08	3.88	3.24	3.51	3.45	3.41
MMX	3.17	5.14	5.61	4.25	5.76	4.68	4.72
RDX	4.07	5.14	6.19	4.95	7.28	5.19	5.63
DEGDN	4.64	6.52	7.81	5.93	7.63	6.16	6.26
MHA	5.14	5.82	5.83	5.95	6.26	5.46	5.75
HC	6.49	8.88	16.03	8.50	16.07	10.15	10.73
ACARADITE I	5.96	4.49	3.65	6.02	4.25	3.90	14.53
TNT	6.91	9.25	24.06	9.18	15.59	10.80	11.24
BTTH	6.95	10.21	16.17	9.66	17.22	10.89	11.97
2,6 DNT	7.65	8.70	11.63	9.51	11.83	8.46	9.47
ACARADITE II	6.86	5.21	4.08	7.21	4.65	4.43	5.07
TMETH	9.28	12.81	25.44	12.82	24.49	14.85	16.01
2,4 DNT	8.03	8.71	11.45	9.47	12.08	8.75	9.38
EC	14.89	15.54	6.48	19.39	14.59	11.55	14.20
MC	14.64	9.39	6.51	15.32	8.84	7.01	8.69
4 NDPA	17.86	15.87	14.84	21.09	22.06	13.72	18.81
NDPA	19.96	18.61	17.42	24.62	26.50	16.95	23.64
2 NDPA	35.06	24.40	18.75	37.27	30.74	19.32	26.91
DPA	36.63	19.36	11.13	38.97	16.29	12.35	18.17

Table 7. Optimization of 5 Micron Dupont Zorbax ODS Column.

	MEOH	ACN	THF	MEOH/ACN	MEOH/THF	ACN/THF	MEOH/ACN/THF
TRIACETIN	3.06	2.88	3.53	3.30	3.50	3.42	3.49
RESORCINOL	3.06	2.88	3.53	3.30	3.50	3.42	3.49
HMX	3.23	5.22	5.48	4.55	5.88	4.88	5.17
PDX	5.20	5.56	6.23	5.64	8.69	5.53	6.39
DEGDN	6.19	7.11	8.01	7.33	8.74	7.07	7.49
MHA	7.33	6.56	6.09	7.74	7.47	6.31	6.68
NG	8.49	10.47	20.13	10.38	22.24	12.43	13.82
ACACADITE I	9.07	5.56	3.52	7.87	4.60	4.06	4.88
TNT	9.67	10.95	29.80	11.64	21.79	13.83	14.75
BTIN	9.74	11.57	19.77	12.04	23.28	13.45	15.43
2,6 DNT	11.83	10.53	13.21	12.14	16.39	10.05	12.35
ACACADITE II	11.53	7.11	3.968	10.03	5.19	4.72	5.65
TMETH	13.31	14.96	30.50	16.90	32.26	18.30	21.60
2,4 DNT	14.15	10.81	13.82	13.12	17.47	10.84	12.62
EC	27.12	20.46	12.32	26.95	20.71	14.31	19.17
MC	31.35	16.11	7.09	25.62	10.28	8.05	10.95
4 NDFA	28.48	18.76	17.76	30.00	31.00	18.27	25.32
NDFA	31.92	24.68	20.52	32.35	39.17	21.98	31.97
2 NDFA	73.83	34840	24.98	59.77	48.35	27.47	40.31
DFA	70.04	34.61	12223	62.94	22.35	15.31	24.15

Table 8. Column Specifications.

Source	Dimensions (MM)	Packing	Partical	Plates
Waters	100 x 5	u-Bondapac C-18	10 Micron Spherical	>5,000
Waters	100 x 5	u-Bondapac C-18	5 Micron Spherical	>9,000
Supelco	250 x 4.6	Supelcosil LC-18	5 Micron Spherical	20,614
E Merck	250 x 4.6	LiChrosorb RP-18	5 Micron Irregular	15,516
Dupont	250 x 4.6	Zorbax ODS	5 Micron Spherical	27,135
PERKIN- ELMER	125 x 4.6	P-E HS-5 C-18	5 Micron Spherical	>8,000

Table 9. N-5 Propellant Analysis.

	NG	DEP	2,2-DN DPA	2,4-DN DPA	LC 2-NDPA	GC 2-NDPA
Arctic	35.74	10.72	--	--	1.792	1.811
Tropic	35.88	10.88	0.090	0.236	1.534	1.716
Desert	35.91	11.13	0.201	0.526	1.388	1.379

Table 10. CHAPARRAL Propellant Analysis.

Comparison of Capillary GC and HPLC

Lot	Condition Aged	Analytical Method	MNA	N-MNA	2-NDPA	4-NDPA	NG	BTTN
E0001	Hot	GC	0.259	0.490	0.244	0.197	12.56	12.86
	Hot	LC	0.263	0.384	0.245	0.209	13.43	12.23
E0003	Ambient	GC	0.564	0.048	0.260	0.229	13.18	13.55
	Ambient	LC	0.609	0.033	0.257	0.230	13.14	13.45
E0003	Hot	GC	0.346	0.380	0.255	0.223	13.04	13.26
	Hot	LC	0.366	0.281	0.244	0.216	13.51	13.62
E0005	Ambient	GC	0.588	0.048	0.260	0.248	12.76	12.79
	Ambient	LC	0.622	0.029	0.256	0.256	12.84	12.74
E0005	Hot	GC	0.112	0.727	0.241	0.211	12.38	14.80
	Hot	LC	0.107	0.624	0.235	0.204	13.00	14.99
E0006	Ambient	GC	0.569	0.042	0.251	0.222	12.81	14.57
	Ambient	LC	0.623	0.022	0.239	0.220	12.75	14.45
E0007	Ambient	GC	0.597	0.039	0.260	0.250	12.95	12.97
	Ambient	LC	0.637	0.024	0.247	0.249	12.80	12.78
E0008	Ambient	GC	0.626	0.023	0.261	0.254	13.26	12.64
	Ambient	LC	0.682	0.022	0.254	0.250	12.94	12.46
E0008	Hot	GC	0.205	0.644	0.340	0.218	13.03	13.65
	Hot	LC	0.218	0.530	0.323	0.210	13.41	13.73
GCU	Ambient	GC	0.615	0.115	0.256	0.261	13.28	14.13
	Ambient	LC	0.659	0.061	0.248	0.239	13.66	14.34

Table 11. Sprint Propellant Analysis.

	Triacetin	Resorcinol	2-Nitrorescinol	NG	2-NDPA
LC	6.657	0.950	0.019	28.734	0.942
GC	6.325	0.901	--	28.400	0.966

Table 12. Foreign Propellant #1 Analysis.

	NG	DPA	MC	NDPA
GC	39.25	0.153	1.104	--
LC	39.41	--	1.345	0.404

Table 13. Foreign Propellant #2 Analysis.

	DEGDN	NG	2,6 DNT + 2,4 DNT	2,4,6 TNT	MC	DPA	NDPA
LC	14.22	17.75	(0.905)	0.375	2.516	0.361	0.249
GC	14.64	17.41	(0.918)	0.356	2.550	0.474	--

Table 14. M-36 Propellant Analysis.

		NG	DNDPA	2-NDPN
GC	Splitless	39.151	3.017	1.950
GC	On-Column	39.134	3.046	1.961
GC	Manual/Split	39.117	3.011	1.969
GC	Auto/Split	39.182	3.002	1.962
GC	Auto/Split	39.012	3.016	1.954
LC		38.626	4.040	1.777

HPLC OPTIMIZATION

DATA ANALYSIS

11.10 AM THU., 24 JAN., 1985

COLUMN : SUPELCO 5u C-18

SAMPLE : N-5 Propellant

THE BEST COMPOSITION IS @

MEOH	ACN	THF
82%	18%	0%

- ◇ RESOLUTION < .714
- RESOLUTION < 1.427
- △ RESOLUTION < 2.141
- ◻ RESOLUTION < 2.676
- ⊙ RESOLUTION = 2.855

MeOH

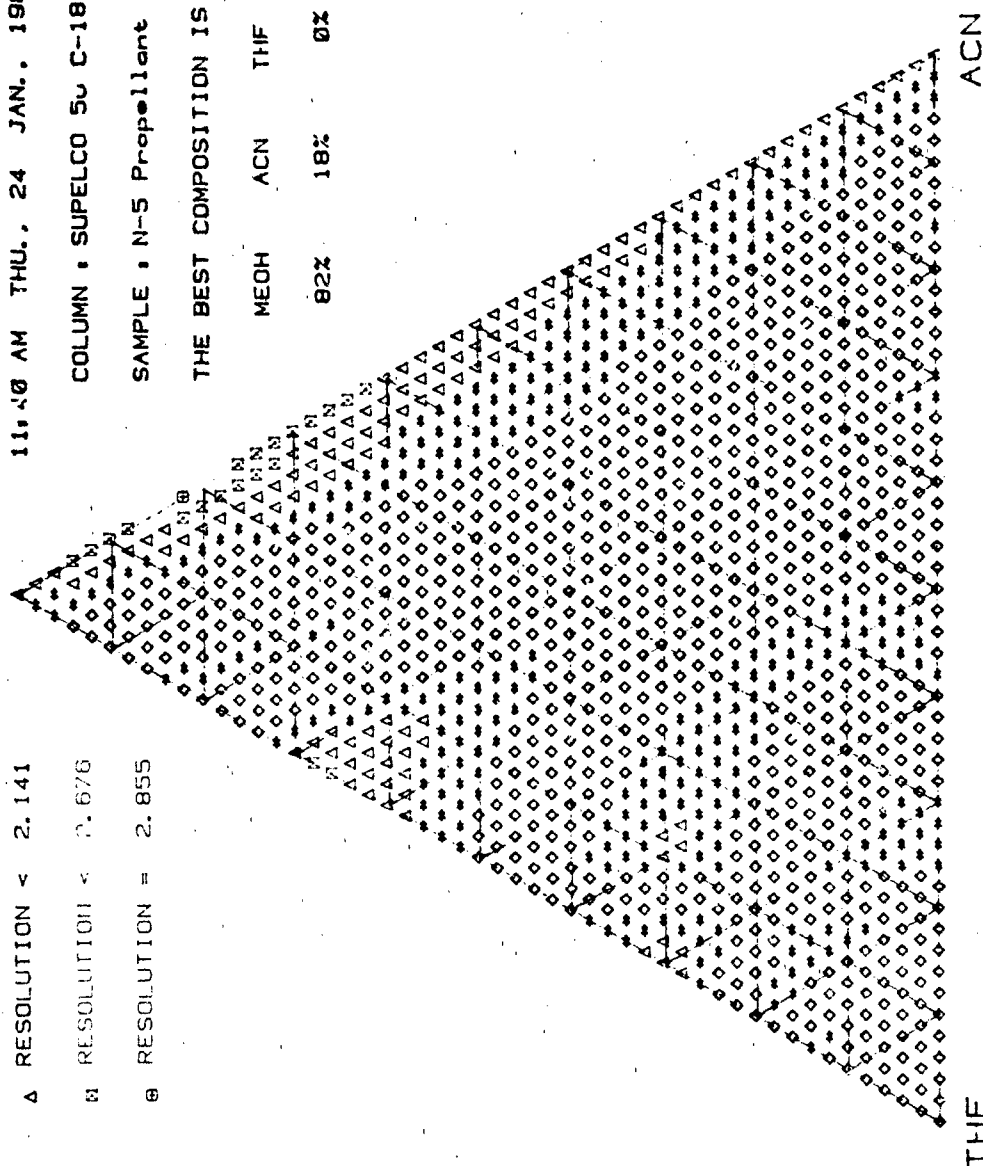


Figure 1. HPLC optimization data analysis.

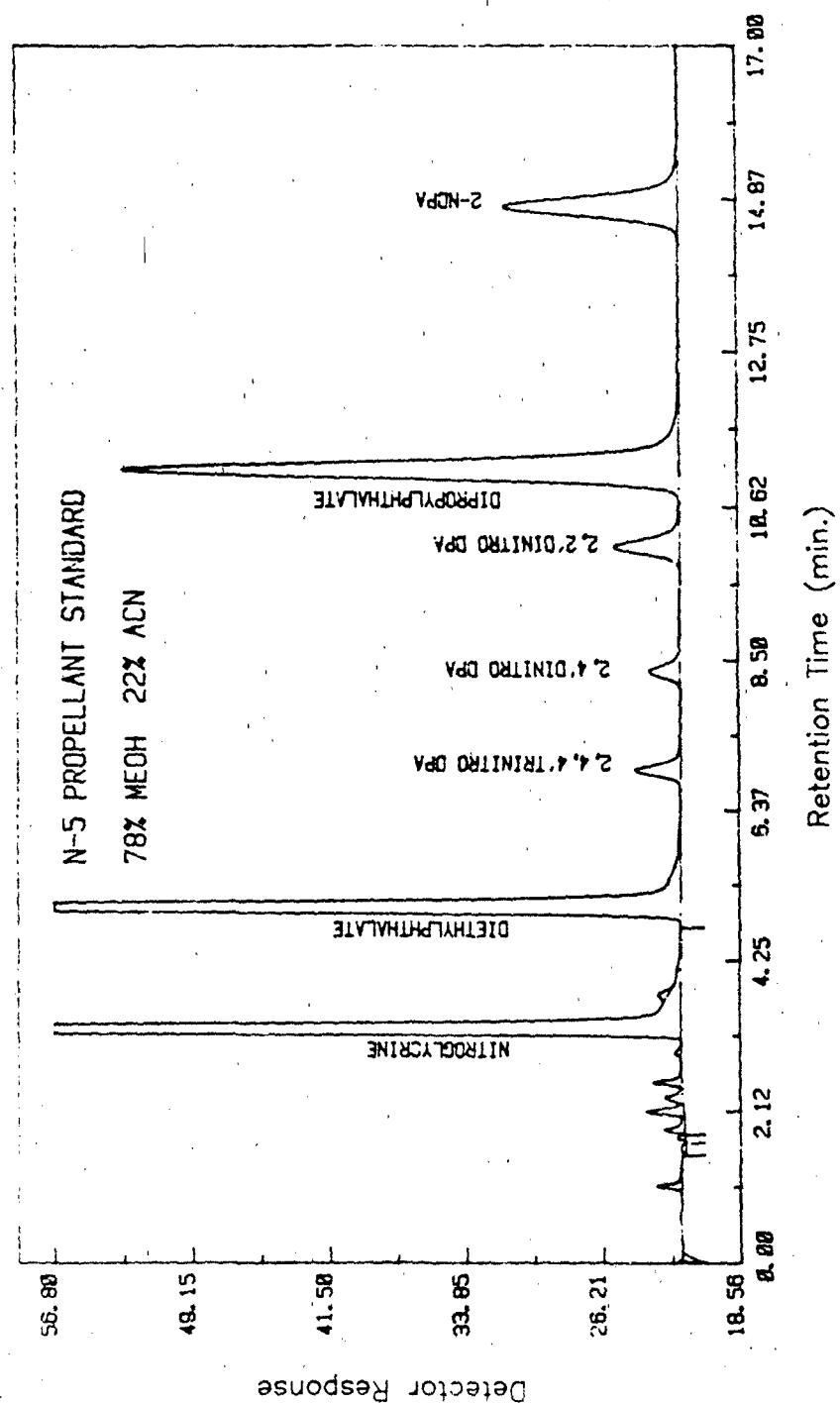


Figure 2. Standard sample.

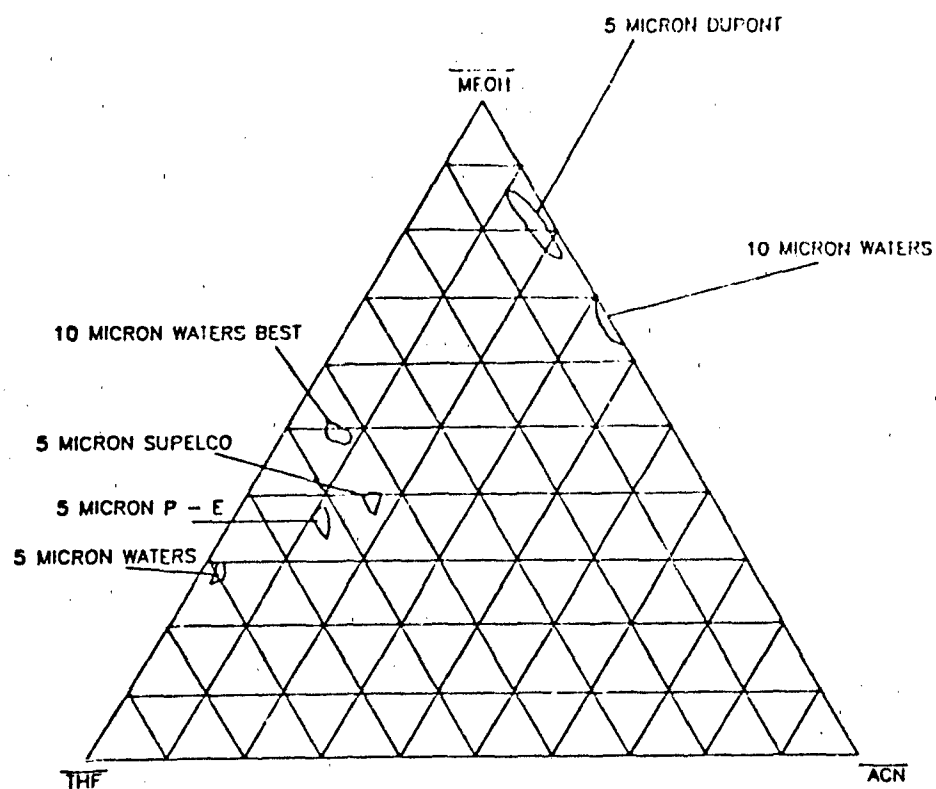


Figure 3. Column composition.

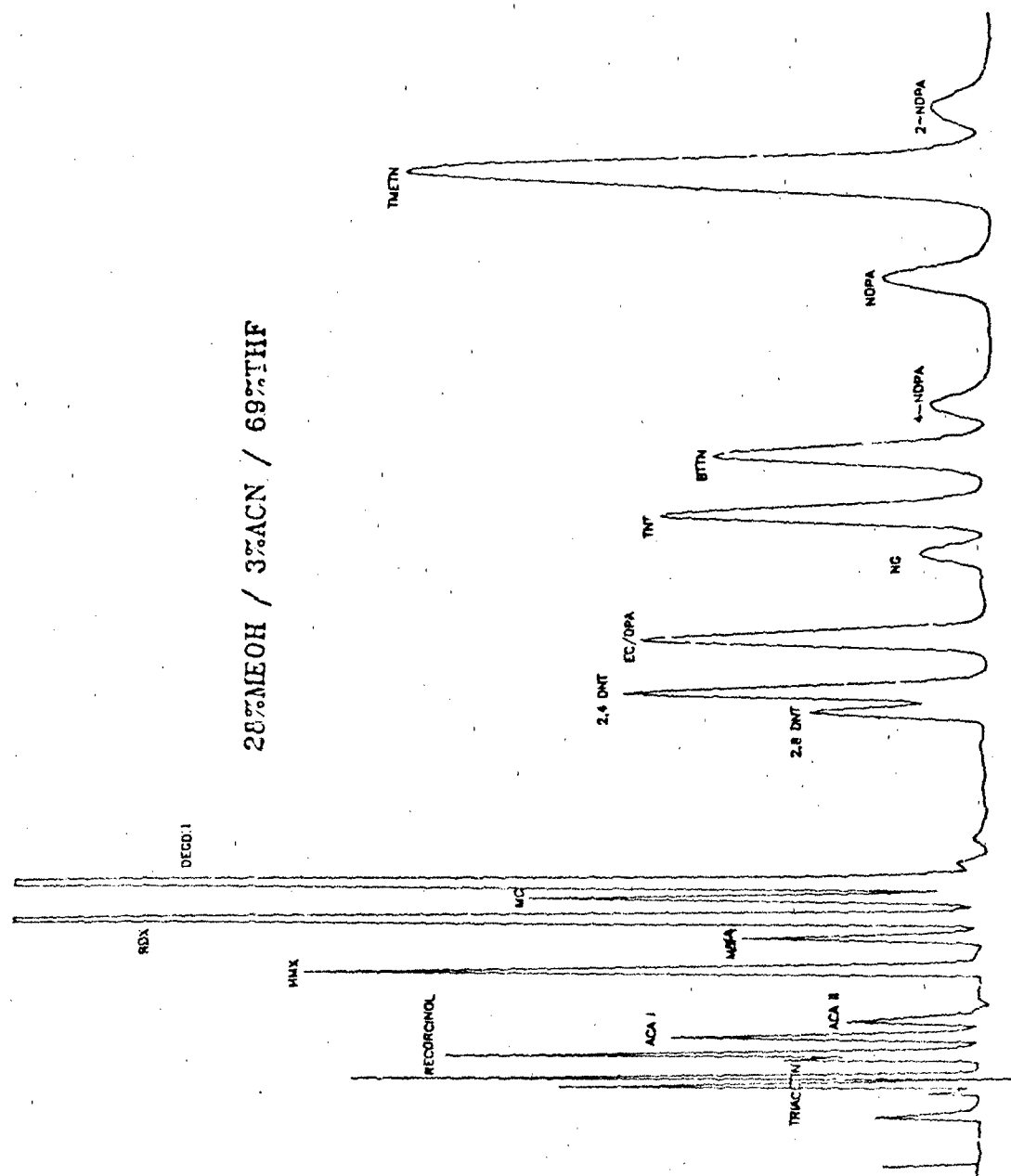


Figure 4. Waters 5 micrometer radial PAC column.

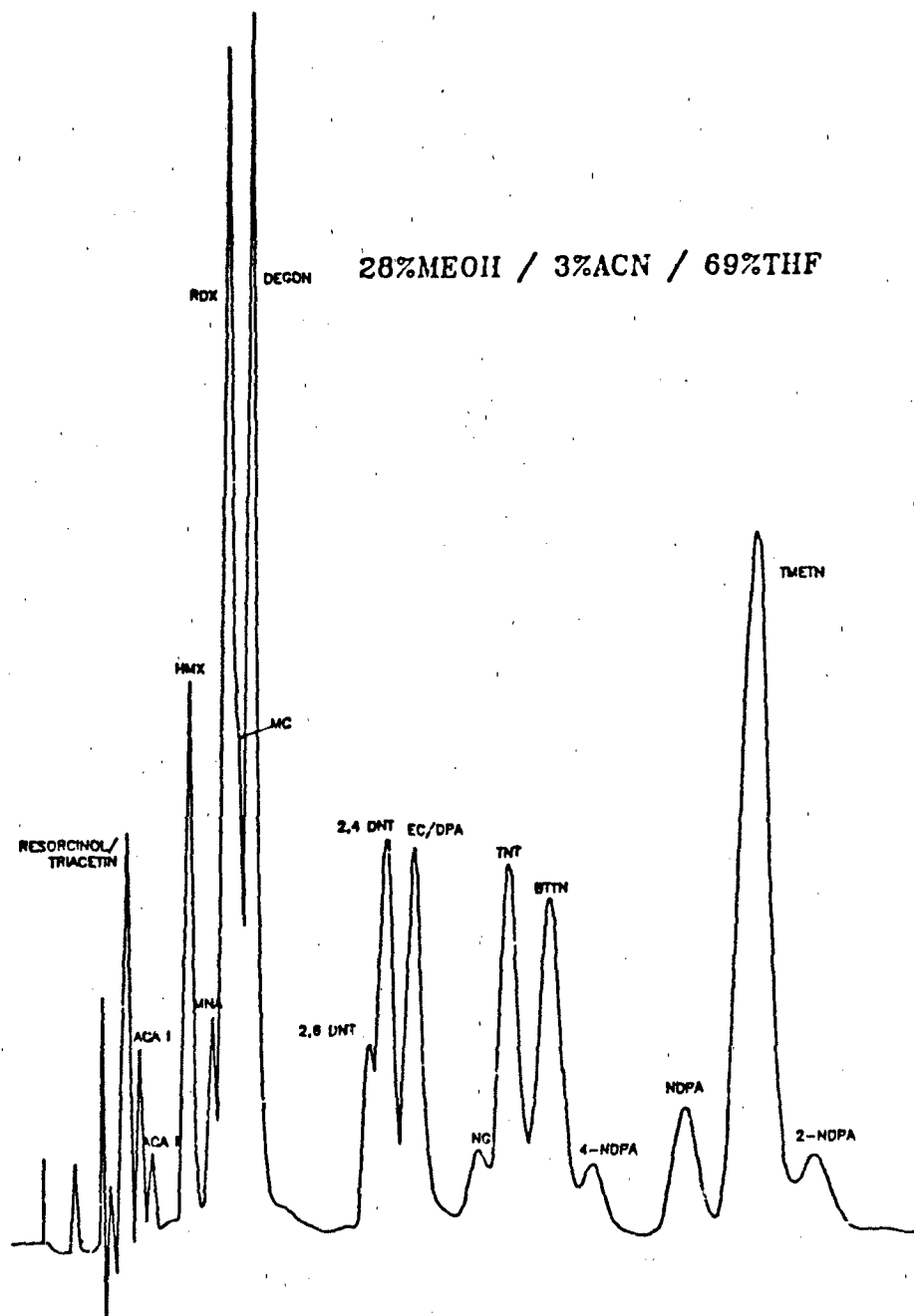


Figure 5: Waters 10 micrometer radial PAC column.

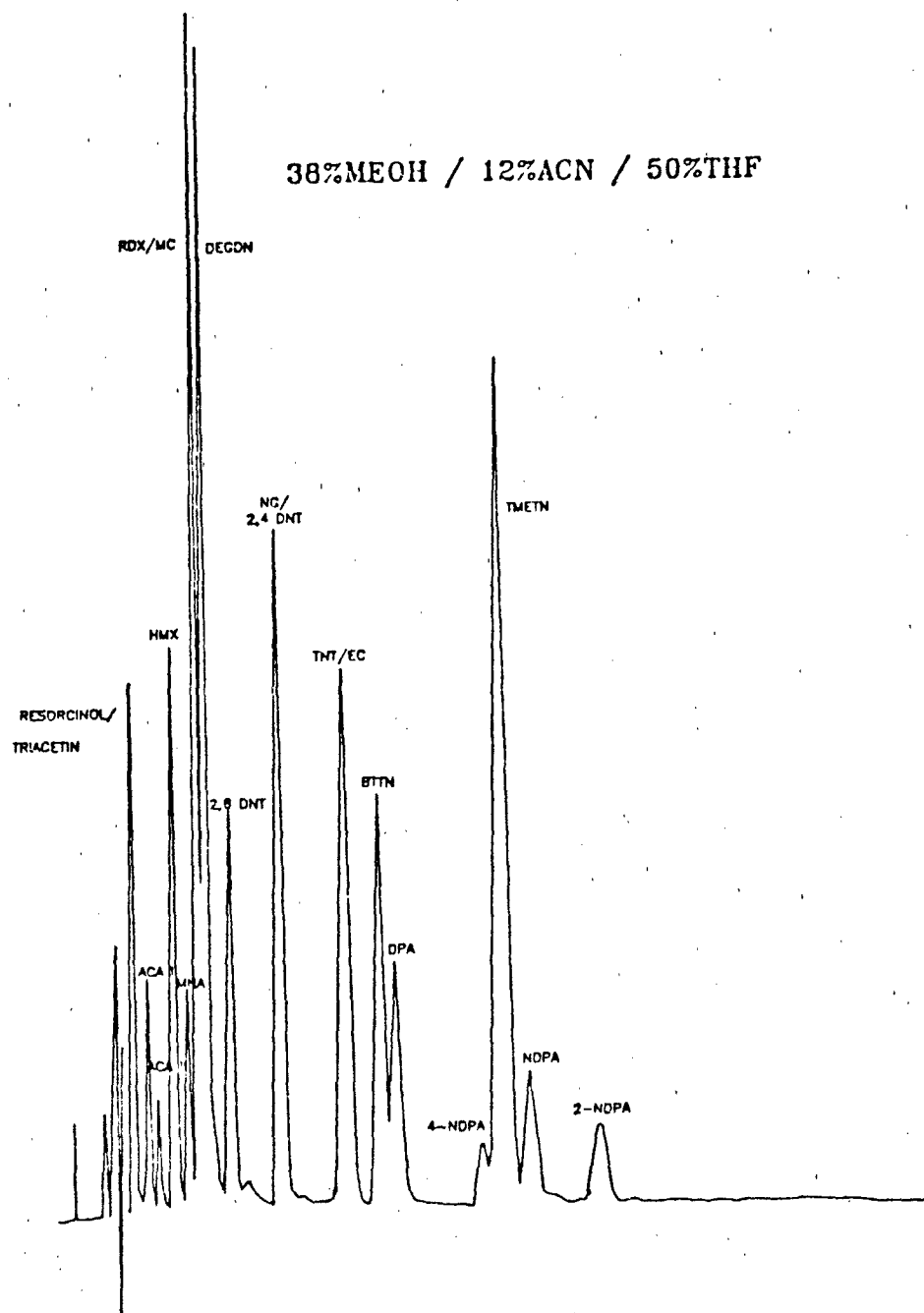


Figure 6. Perkin Elmer HS-5C-18 column.

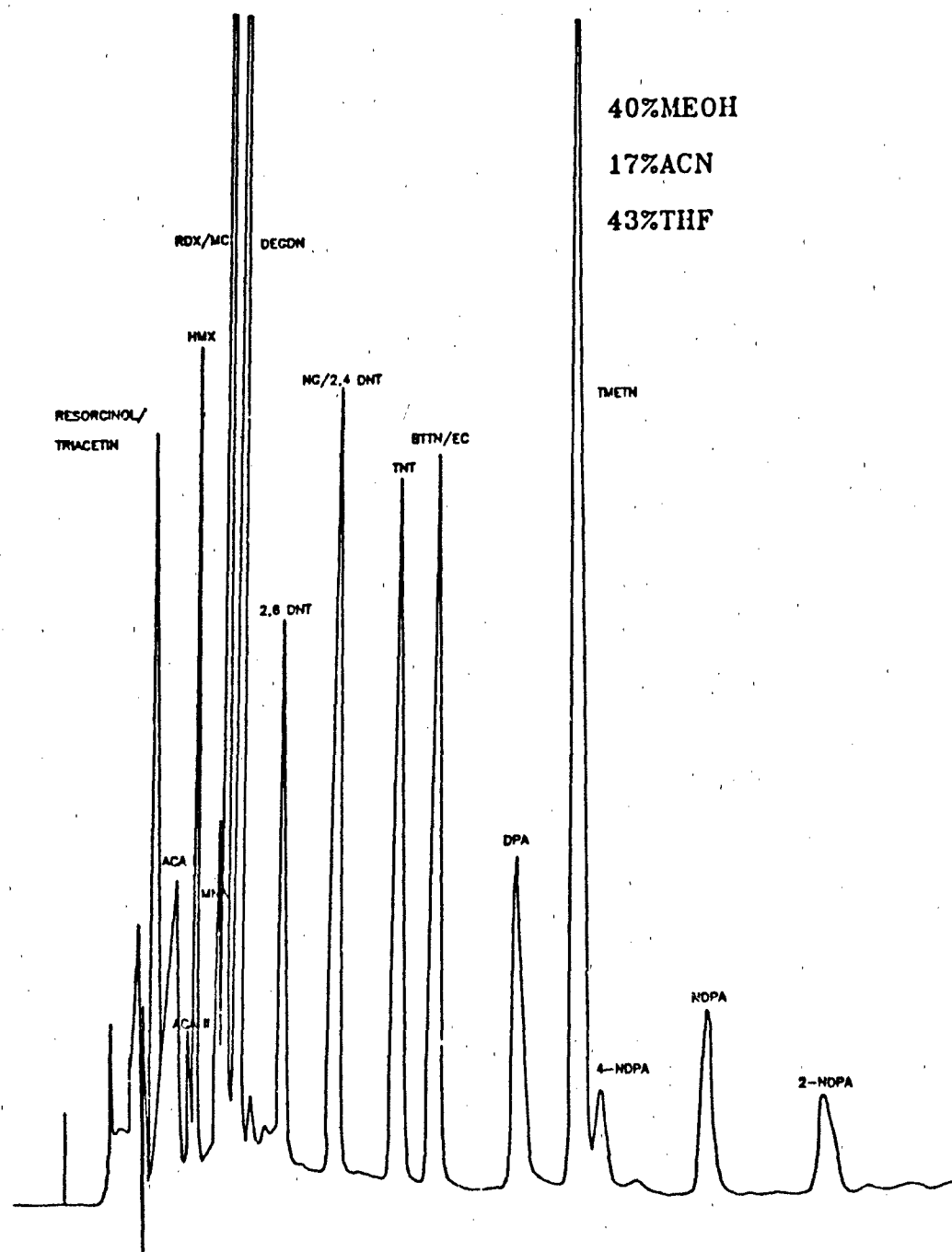


Figure 7. Suplecasil LC-18 column.

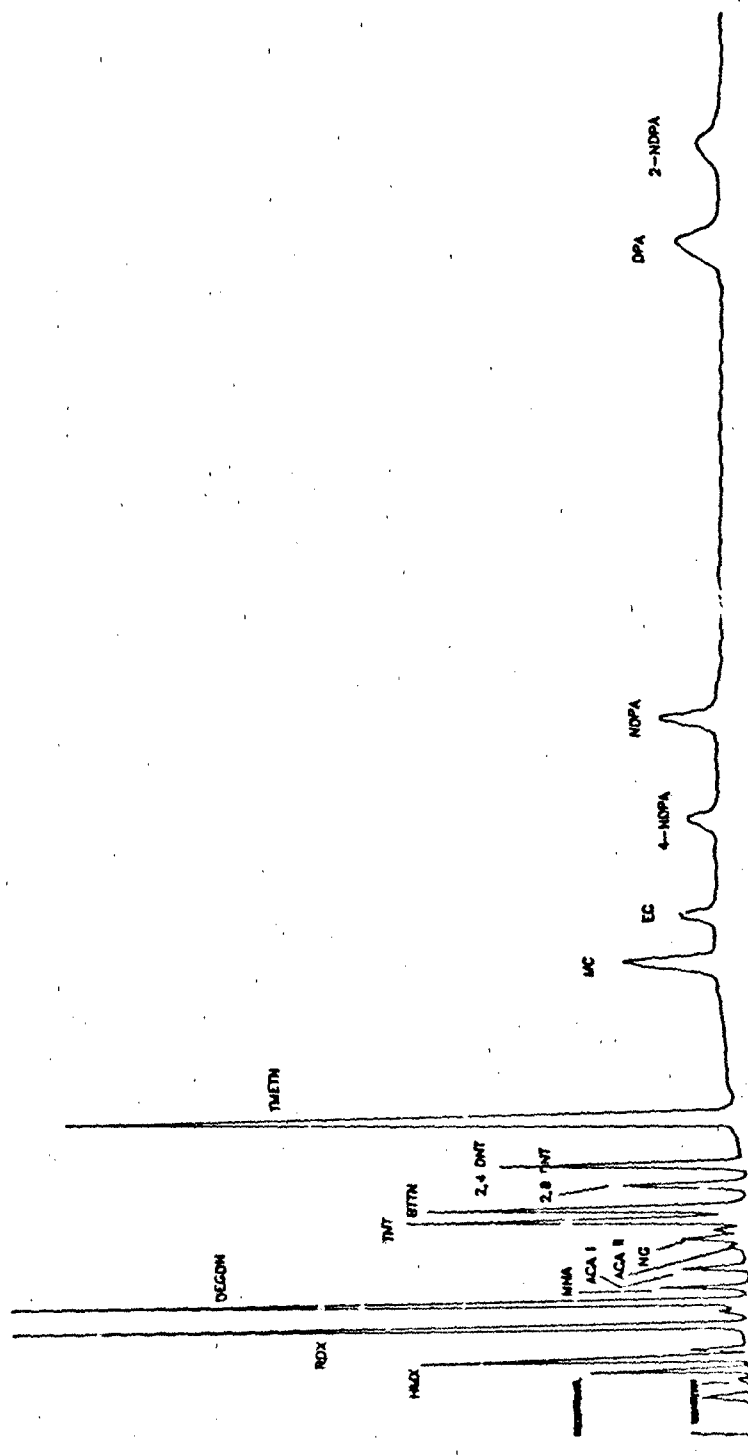


Figure 8. Dupont ODS column.

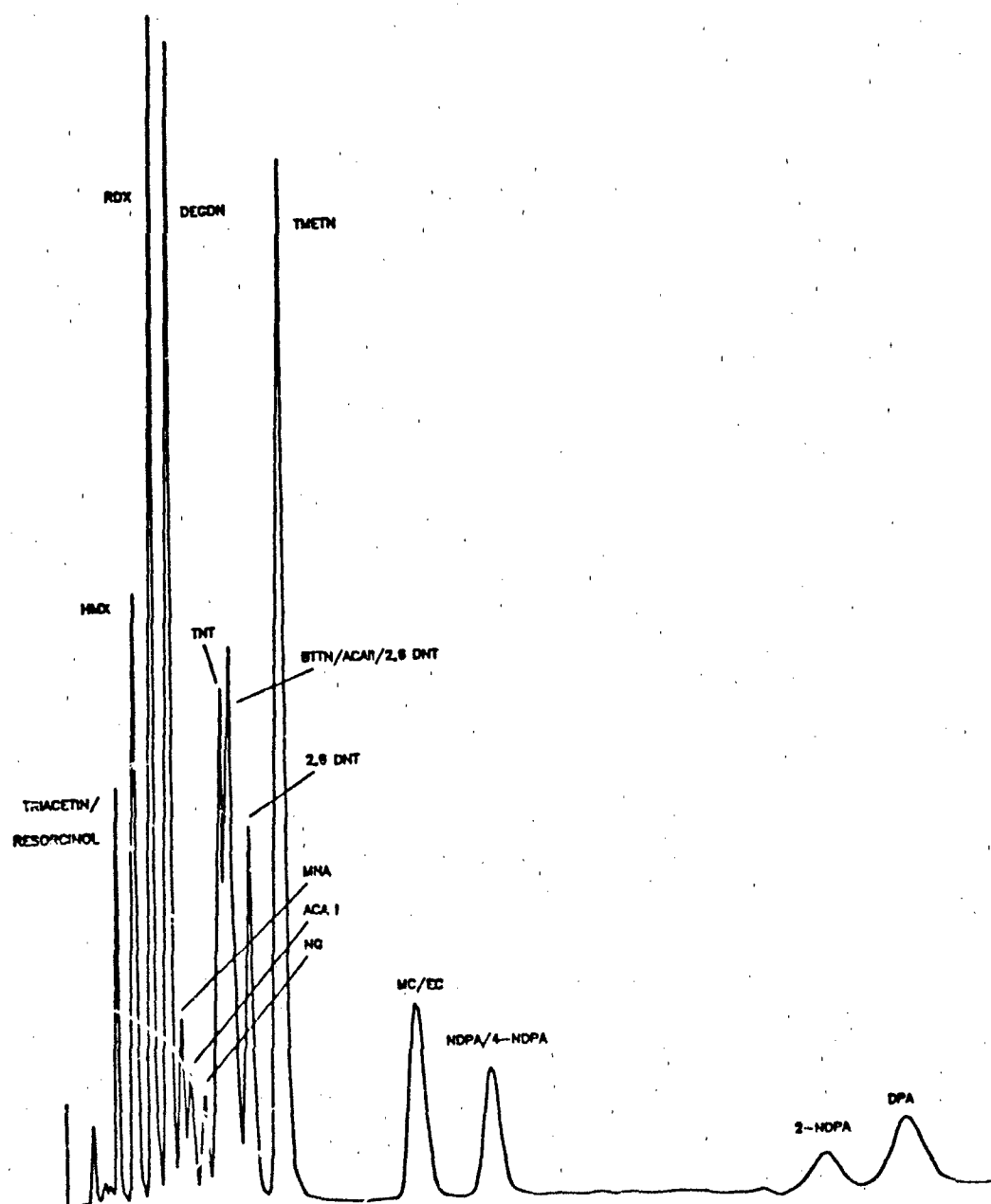


Figure 9. Waters 10 micrometer radial Pac column.

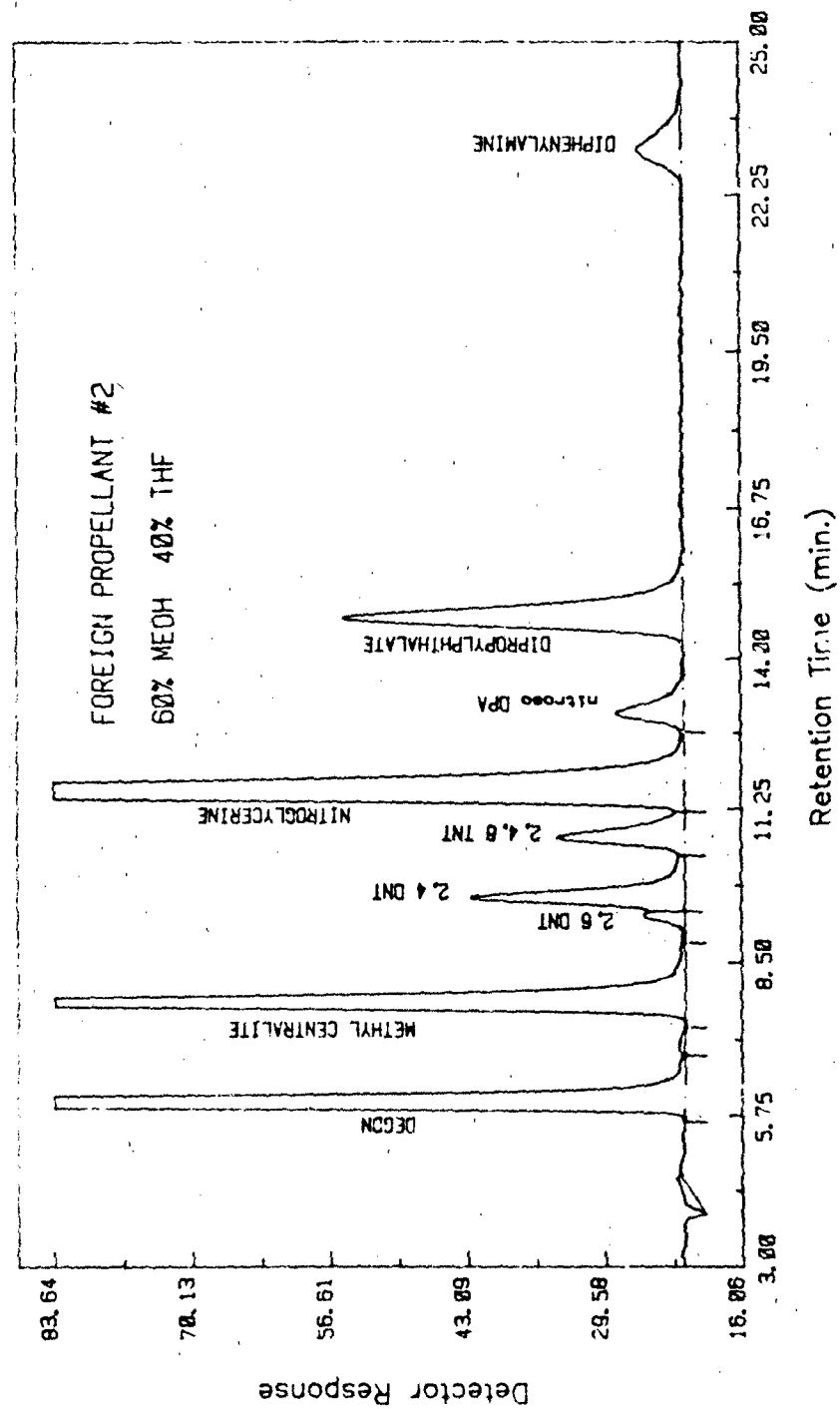


Figure 10. Foreign propellant #2.

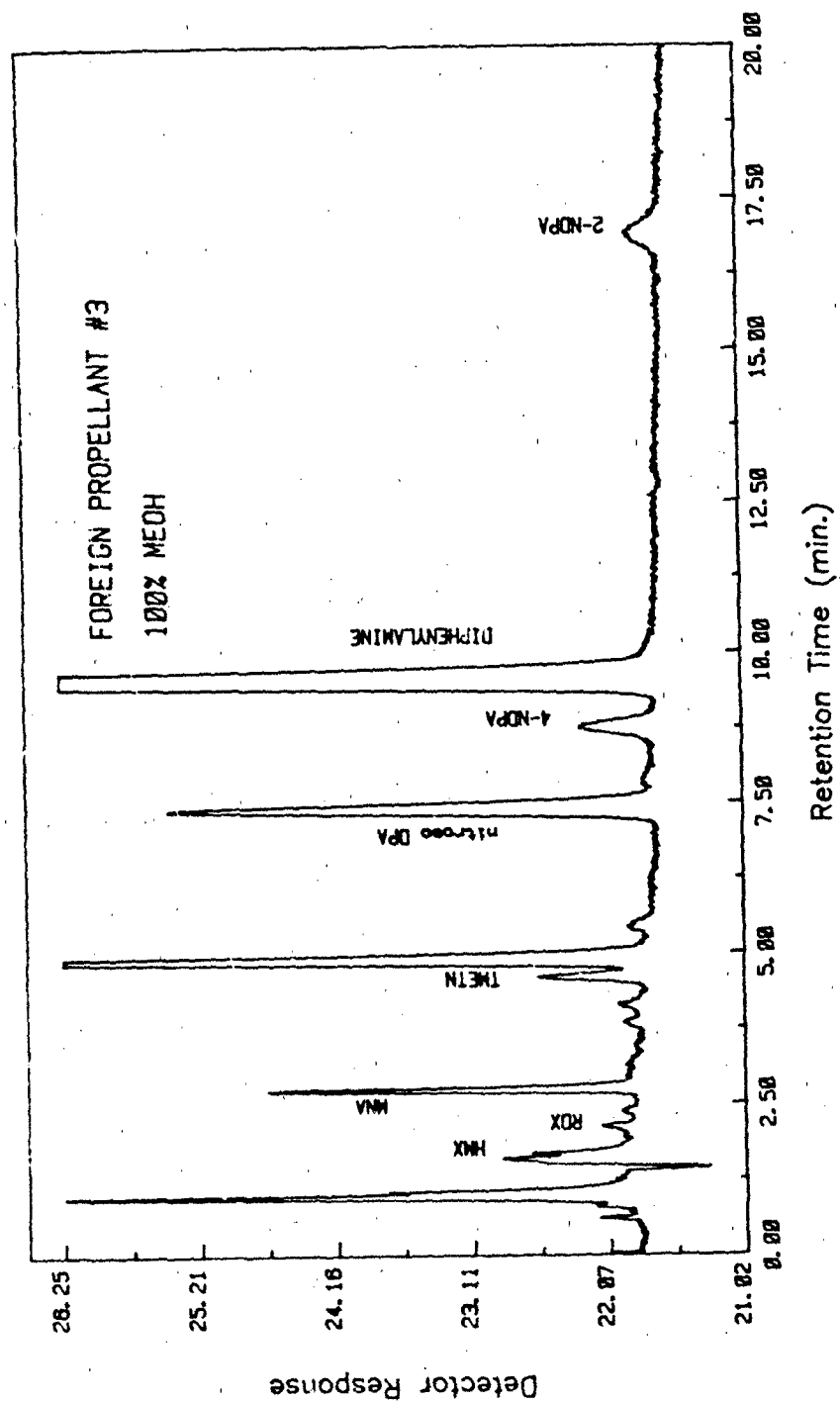


Figure 11. Foreign propellant #3.

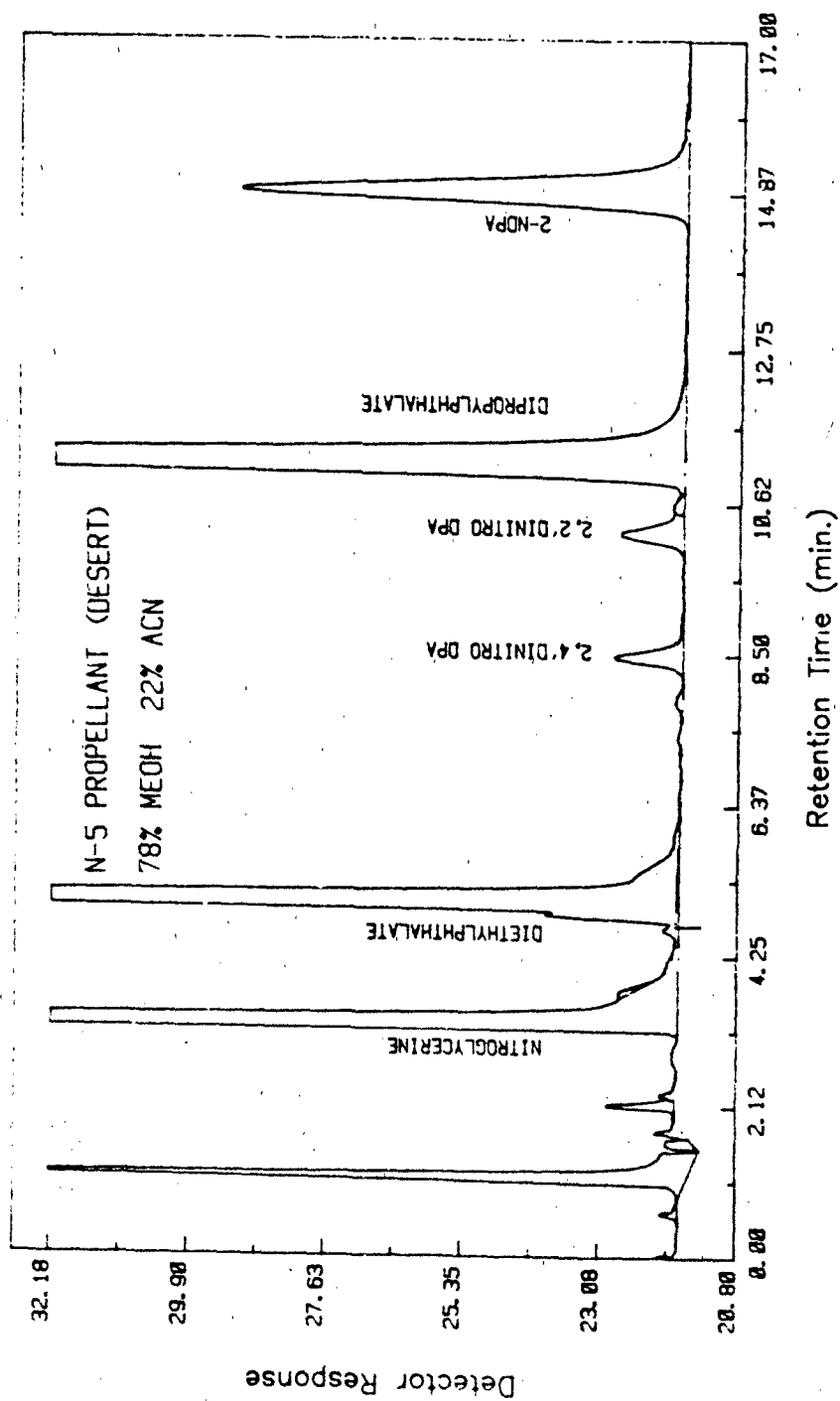


Figure 12. N-5 propellant (desert).

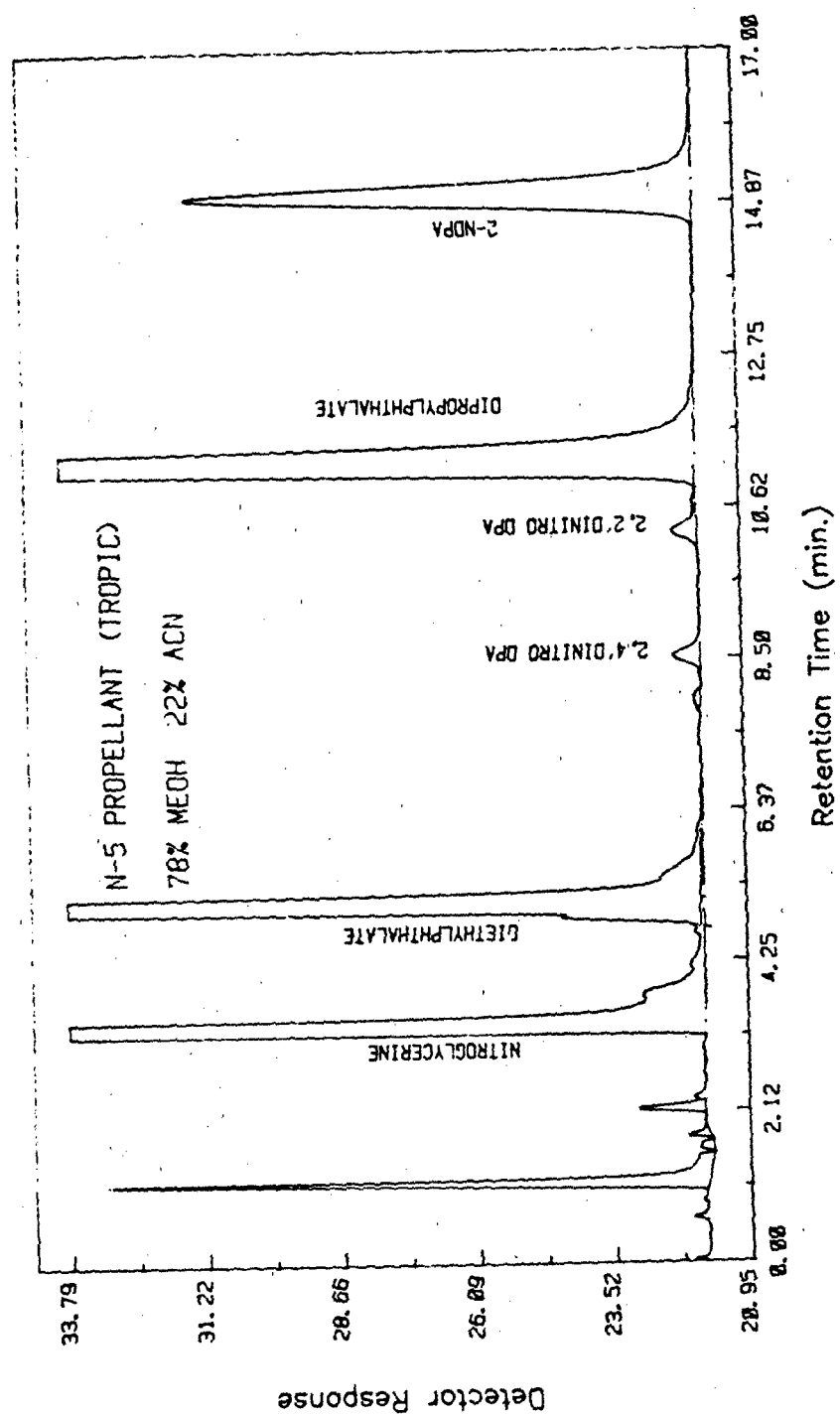


Figure 13. N-5 propellant (tropic).

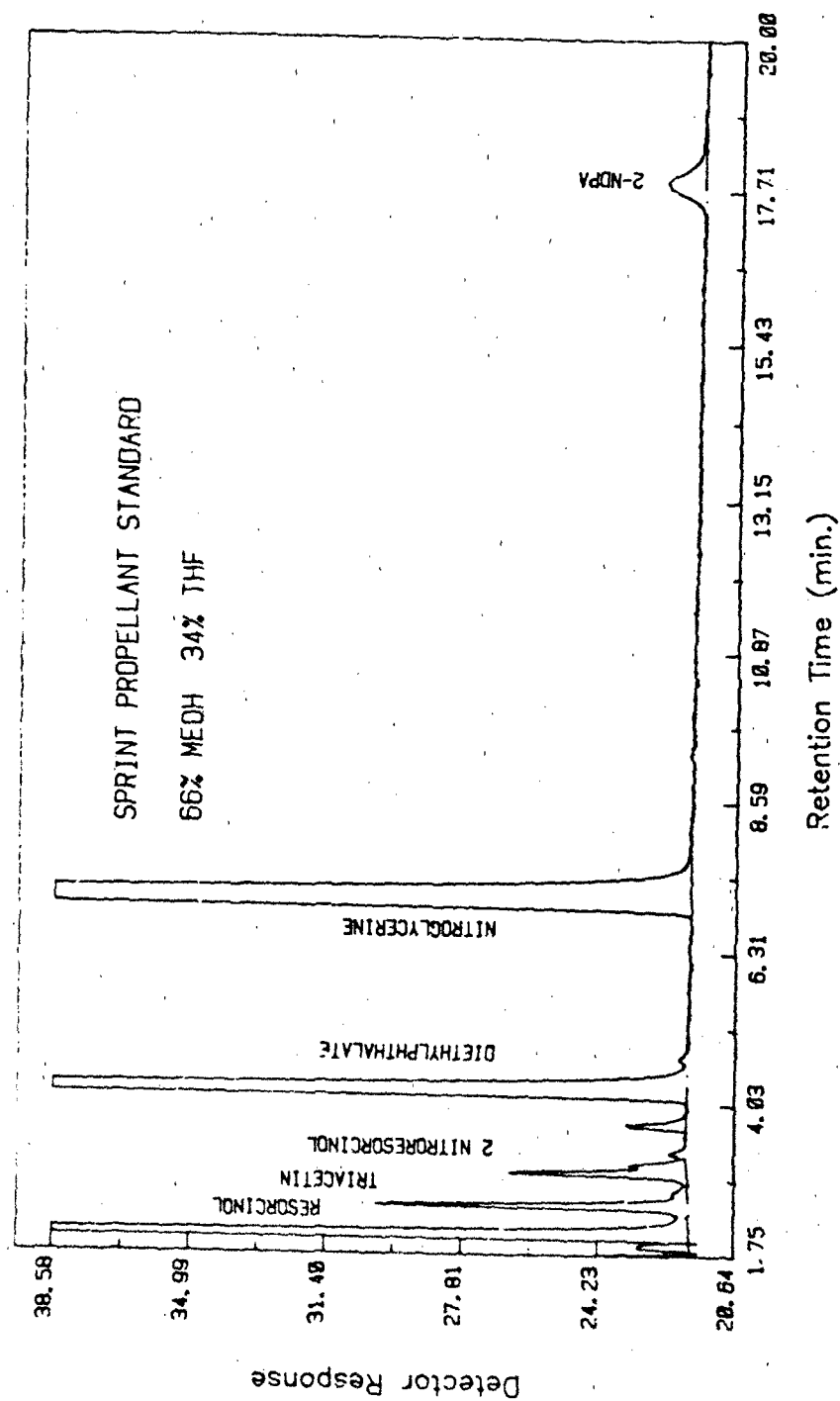


Figure 14. SPRINT propellant standard.

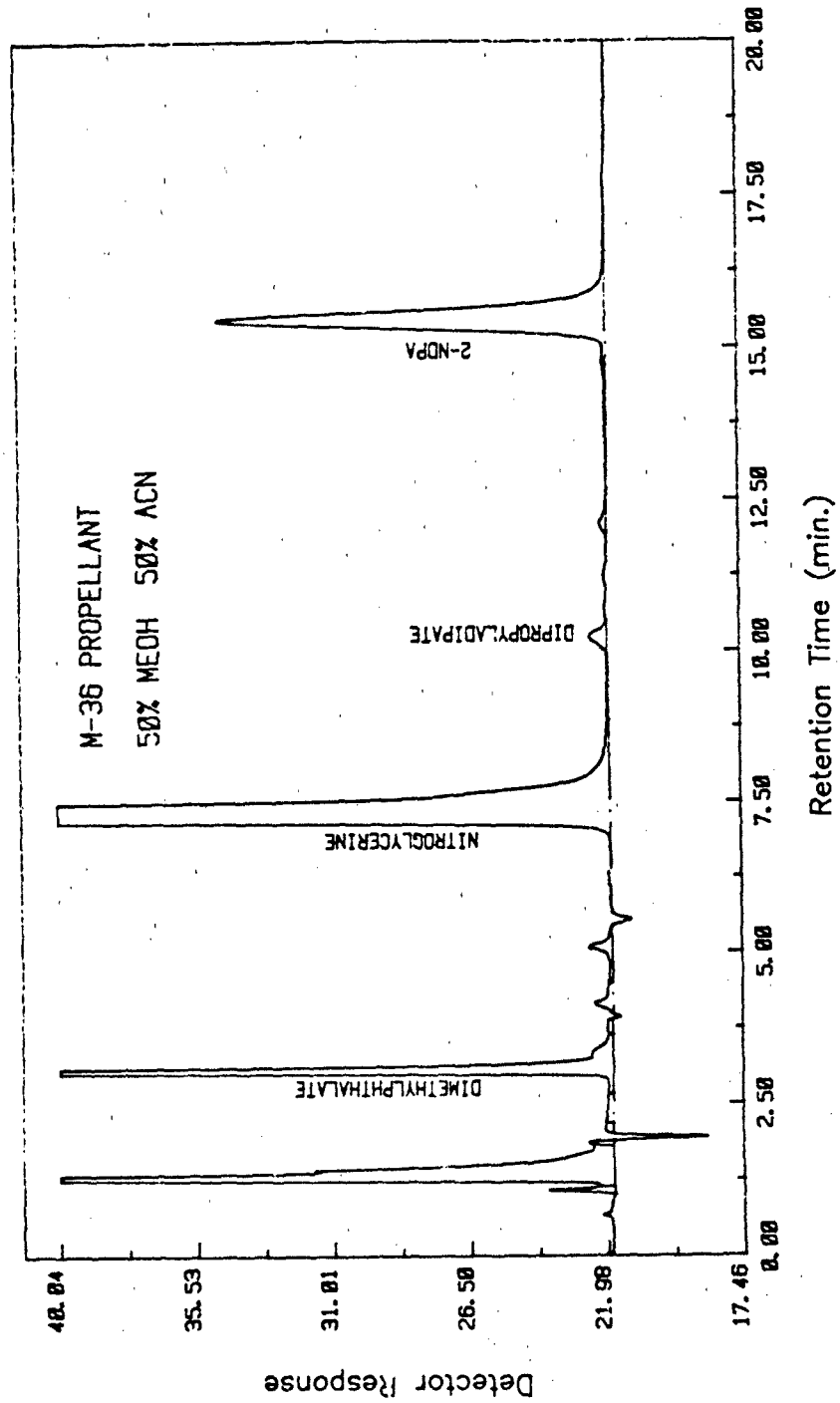


Figure 15. M-36 propellant.

LIST OF ABBREVIATIONS

HPLC	High Pressure Liquid Chromatography
GC	Gas Chromatography
MeOH	60% Methanol Water Mixture
ACN	50% Acetonitrile Water Mixture
THF	42% Tetrahydrofuran Water Mixture
DPA	Diphenylamine
NDPA	Nitrosodiphenylamine
2-NDPA	2-Nitrodiphenylamine
4-NDPA	4-Nitrodiphenylamine
2,2-DNDPA	2,2-Dinitrodiphenylamine
2,4'-DNDPA	2,4'-Dinitrodiphenylamine
2,4,4'-TNDPA	2,4,4'-Trinitrodiphenylamine
2,4-DNT	2,4-Dinitrotoluene
2,6-DNT	2,6-Dinitrotoluene
2,4,6-TNT	2,4,6-Trinitrotoluene
BTTN	1,2,4-Butanetrioltrinitrate
DEGDN	Diethyleneglycoldinitrate
DNDPA	Di-n-propyladipate
MNA	N-Methylaniline
NMNA	N-Nitroso-n-methylaniline
NG	Nitroglycerine
TIMETN	2-hydroxymethyl-2-methyl-1,3-propanedioltrinitrate

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APPENDIX A
DATA ACQUISITION

The analog out put from the detector was digitized and sent to a Hewlett-Packard Laboratory Automation System. The data was then integrated in the normal manner. A post run basic language program "PEAKWT" listed below was then automatically run. This program examines the data file for the last chromatograph. For each peak integrated, the program finds the maximum amplitude. It then samples the data points before and after the peak until it has a corrected value of 10% of the maximum. The retention time of these points are then subtracted and reported as the width of that peak.

The 10% was arbitrarily selected because it is more reproducible and does not confuse a tailing peak with a very broad peak. This system gives more meaningful results when all ingredients are well separated and of approximately the same amount.

In Line 190 A\$ will be returned string with the name of the raw data file and B\$ the name of the processed data file. In line 240 and 250 W is the width of a data slice and N is the number of peaks integrated. In line 290 T(I) and P(I) are the retention time and amount of peak I. Line 320 sets the pointer to the first record containing extended data. Line 350 reads the height, start, baseline at start, end, and baseline at end of each peak I. The Loop in 480 to 540 finds the 10% levels at the front and end of each peak I.

```

100 REM *****
110 REM *** BASIC PROGRAM "PEAKWT" ***
120 REM *** CALCULATES PEAK WIDTH AT 10% OF THE PEAK HEIGHT. ***
130 REM *** TO CALCULATE AT A DIFFERENT FRACTION OF THE PEAK ***
140 REM *** HEIGHT CHANGE LINES 360 AND 370. ***
150 REM *** CREATED BY J. CARVER 7 JUNE 1984 ***
160 REM *****
165 COM BS[10],CS[10],MS[10],RS[10],PS[10],N[10],D[2,4],Z[4]
170 DIM NS[26],A[35],B[35],C[35],E[35],H[35],P[35],S[35],T[35],U[35]
180 FILES *,*
190 TELL AS,BS,CS,E
200 ASSIGN BS,1,E,F
210 ASSIGN CS,2,E
220 ON END #1 THEN 1000
230 ON END #2 THEN 1000
240 READ #1,NS,X,X,NS,X,NS,X,X,X,X,NS,NS,NS,NS,NS,X,X,W
250 READ #1,1,NS,X,X,NS,X,NS,X,X,X,X,NS,NS,NS,NS,NS,X,X,X,X,N
260 LET N=ABS(N)
270 READ #1,2
280 FOR I=1 TO N
290 READ #1:T[I],X,X,X,X,P[I],NS
300 NEXT I
310 LET R4=1
320 READ #1,(F/2)+2
330 READ #2,2
340 FOR I=1 TO N
350 READ #1:H[I],S[I],X,B[I],E[I],X,U[I],NS
360 LET A[I]=(H[I]*.1)+B[I]
370 LET C[I]=(H[I]*.1)+U[I]
380 LET S[I]=S[I]*60
390 LET E[I]=E[I]*60
400 LET T[I]=T[I]*60
410 LET R1=INT(S[I]/W)
420 LET R2=INT(E[I]/W)
430 LET R3=INT(T[I]/W)
440 FOR K=R4 TO R1-1
450 READ #2,N2
460 NEXT K
470 IF R4 >= R1 THEN LET R1=R4
480 FOR K=R1 TO R2
490 READ #2,N2
500 IF K>R3 GOTO 530
510 IF N2 >= A[I] AND INT(S[I]/W)=R1 THEN LET S[I]=K*W
520 GOTO 540
530 IF N2 <= C[I] AND INT(E[I]/W)=R2 THEN LET E[I]=K*W
540 NEXT K
550 LET R4=R2+1
560 NEXT I
1000 PPINT
1010 PRINT
1020 PRINT "PROCESSED FILE ";BS
1030 PRINT
1040 PRINT TAB(30);"10% OF HEIGHT"
1050 PPINT "PEAK RT","AREA A ","PEAK WIDTH (SEC)"
1060 PRINT
1070 FOR I=1 TO N
1080 LET W=(E[I]-S[I])
1090 LET T[I]=T[I]/60
1100 PRINT T[I],P[I],W
1110 NEXT I
1120 PPINT
1130 PPINT
1140 PPINT
1150 END

```

APPENDIX B
DATA REDUCTION BASIC LANGUAGE VERSION

The basic language version of the data reduction was designed to output to a Hewlett-Packard 2648 graphics terminal with a 9876 A graphics printer attached as a "C" device.

Line 145 to 155 in program XLDB6A open previously created data files to the program. These 7 files contain the retention data from the 7 solvent compositions. This program can enter new data or change old data in these files. It can not create the files. This must be done separately by the user. When the files are satisfactory, the user can list the data input as well as the resolution for pairs of peaks in each solvent. A chromatographic optimization function, as discussed by Synder [21], is also calculated. The program then calls XLDB6B for further calculations and plotting lines of resolution for each peak pair on the graphics terminal.

```

10  COM M[2],T[7,35]
100 REM *****
105 REM *****LIQUID CHROMATOGRAPHIC OPTIMIZATION*****
110 REM ***** XLDB6A **R.RADKE / J.CARVER *****
115 REM *****AUG/SEPT 1981*****
120 REM *****
125 LET M1=7
130 LET M2=8
135 DIM ES[1],S[7],V[7],W[7,35],K[7,35],R[7,35]
140 CHRS 27,ES
145 FILES FIRST::19,SECOND::19,THIRD::19
150 FILES FOURTH::19,FIFTH::19,SIXTH::19
155 FILES SEVENTH::19
160 REM GET SOLVENTS
165 PRINT
170 PRINT " ENTER THE NUMBER OF SOLVENTS (1-7)  <0=EXIT>"
175 INPUT M1
180 IF M1=0 THEN 9999
185 IF M1<1 THEN 170
190 IF M1>7 THEN 170
195 LET M[1]=M1
200 REM GET PEAKS
205 PRINT " ENTER THE NUMBER OF PEAKS PER SOLVENT (2-35)  <0=EXIT>"
210 INPUT M2
215 IF M2=0 THEN 9999
220 IF M2<2 THEN 205
225 IF M2>35 THEN 205
230 LET M[2]=M2
235 REM LOOP TO GET DATA
240 FOR J=1 TO M1
245 PRINT " ENTER THE RETENTION TIME (MIN) FOR SOLVENT ";J;" <0=EXIT>"
250 INPUT V[J]
255 IF V[J]=0 THEN 9999
260 LET V[J]=V[J]*60
265 PRINT " ENTER THE AVERAGE BAND WIDTH <SEC> FOR ALL PEAKS OF SOLVENT ";J
270 INPUT A
275 FOR I=1 TO M2
280 PRINT " ENTER THE RETENTION TIME (MIN) FOR PEAK ";I;" <0=EXIT>"
285 INPUT T[J,I]
290 IF T[J,I]=0 THEN 9999
295 LET T[J,I]=T[J,I]*60
300 LET W[J,I]=A
305 LET K[J,I]=(T[J,I]-V[J])/V[J]
310 NEXT I
315 NEXT J
320 GOSUB 605
325 REM CALCULATE THE RESOLUTION
330 GOSUB 6000
335 FOR J=1 TO M1
340 FOR I=1 TO M2-1
345 FOR K=I+1 TO M2
350 LET A=(T[J,K]-T[J,I])/(.5*(W[J,K]+W[J,I]))
355 PRINT #J;A
360 NEXT K
365 NEXT I
370 NEXT J
375 REM *** CALCULATE RESPONSE FUNCTION ***
380 PRINT " ENTER DESIRED RESOLUTION  <0=GO TO ORM>"
385 INPUT R9
390 IF R9=0 THEN CHAIN "XLDB6B"

```

```

395 IF R9<0 THEN 380
400 GOSUB 6000
405 FOR J=1 TO M1
410 LET S(J)=0
415 FOR I=1 TO M2-1
420 FOR K=I+1 TO M2
425 READ R9;A
430 LET Z=A/R9
435 IF Z<= 0 THEN 445
440 LET S(J)=S(J)+LN(Z)
445 NEXT K
450 NEXT I
455 NEXT J
460 REM *** OUTPUT TABLE ***
465 GOSUB 6000
470 FOR J=1 TO M1
475 OUTDVC "C1",E
480 PRINT ES"U"
485 PRINT " RETENTION TIME FOR SOLVENT ";J;" IS ";(V(J)/60);" MIN"
490 PRINT
495 PRINT
500 PRINT " PEAK"," RETEN TIME"," BAND WIDTH"," CAPACITY"
505 FOR I=1 TO M2
510 PRINT TAB(3);I;TAB(19);(T(J,I)/60);TAB(34);W(J,I);TAB(47);K(J,I)
515 NEXT I
520 PRINT
525 PRINT
530 PRINT " FOR PEAKS"," RESOLUTION"
535 PRINT
540 FOR I=1 TO M2-1
545 FOR K=I+1 TO M2
550 READ R9;A
555 PRINT TAB(3);I;"- ";K;TAB(17);A
560 NEXT K
565 NEXT I
570 PRINT
575 PRINT
580 PRINT " CHROMATOGRAPHIC OPTOMIZATION FUNCTION (COF) FOR SOLVENT ";J;
585 PRINT " IS ";S(J)
590 NEXT J
595 OUTDVC "T",E
600 CHAIN "XLD063"
605 REM PRINT INPUT
610 PRINT
615 PRINT
620 PRINT TAB(8)"SOLVENT-1 SOLVENT-2 SOLVENT-3 SOLVENT-4 SOLVENT-5";
625 PRINT TAB(58)"SOLVENT-6 SOLVENT-7"
630 PRINT TAB(8)"-----";
635 PRINT TAB(58)"-----"
640 PRINT "TO";
645 FOR X=1 TO M1
650 PRINT TAB(X*10-2);V(X)/60;
655 NEXT X
660 PRINT
665 PRINT "WIDTH";
670 FOR X=1 TO M1
675 PRINT TAB(X*10-2);W(X,1);
680 NEXT X
685 PRINT
690 FOR Y=1 TO M2

```

```

695 PRINT "RT="Y;
700   FOR X=1 TO M1
705   PRINT TAB(X*10-2);T[X,Y]/60;
710   NEXT X
715 PRINT
720 NEXT Y
725 PRINT TAB(8);"-----";
730 PRINT TAB(58);"-----"
735 PRINT
740 PRINT "ARE THERE ANY CHANGES";
745 INPUT CS
750 IF CS[1,1]="Y" THEN 1000
755 RETURN
1000 REM MAKE CHANGES
1005 PRINT "WHICH SOLVENT (1-";M1;")";
1010 INPUT J
1015 PRINT "TO=";V[J]/60;" THE NEW VALUE IS";
1020 INPUT V[J]
1025 LET V[J]=V[J]*60
1030 PRINT "WIDTH =" ;W[J,1];" THE NEW VALUE IS";
1035 INPUT A
1040   FOR X=1 TO M2
1045   LET W[J,X]=A
1050   NEXT X
1055 PRINT "WHICH PEAK NEEDS TO BE CHANGED (1-";M2;")";
1060 INPUT I
1065 PRINT "PEAK ";I;"HAS A RETENTION TIME OF ";T[J,I]/60;" THE NEW VALUE IS";
1070 INPUT T[J,I]
1075 LET T[J,I]=T[J,I]*60
1080 LET K[J,I]=(T[J,I]/V[J])-1
1085 PRINT
1090 PRINT "ANY MORE CHANGES FOR SOLVENT ";J;
1095 INPUT CS
1100 IF CS[1,1]="Y" THEN 1055
1105 PRINT "ANY CHANGES FOR ANOTHER SOLVENT";
1110 INPUT CS
1115 IF CS[1,1]="Y" THEN 1005
1120 GOSUB 610
1125 GOTO 325
6000 REM *** RESET POINTERS ***
6005 READ #1,1
6010 READ #2,1
6015 READ #3,1
6020 READ #4,1
6025 READ #5,1
6030 READ #6,1
6035 READ #7,1
6040 RETURN
9999 END

```

```

10  COM A[2],T[7,35]
100  REM *****
105  REM *****LIQUID CHROMATOGRAPHIC OPTINIZATION*****
110  REM ***** XLDB6B **R.RADKE / J.CARVER *****
115  REM *****AUG/SEPT 1981*****
120  REM *****
125  CHRS 27,FS
130  DIM ES[1],YS[10],ZS[26],B[35],C[35]
135  LET YS="0123456789"
140  LET ZS="          : AM"
145  LET M[1]=M[3]=M[5]=M[7]=M[8]=M[10]=M[12]=31
150  LET M[4]=M[6]=M[9]=M[11]=30
155  LET M[2]=28
160  FILES FIRST::19,SECOND::19,THIRD::19
165  FILES FOURTH::19,FIFTH::19,SIXTH::19
170  FILES SEVNTN::19,RESOLV::19,HOLDER::19
175  READ #8,1
180    FOR I1=0 TO 100 STEP 2
185      FOR J1=0 TO 100-I1 STEP 2
190        PRINT #8;1.00000E+38
195      NEXT J1
200    NEXT I1
2000  REM *** ORM SUBROUTINE ***
2005  OUTDVC "T*",E
2010  LET M1=A[1]
2015  LET M2=A[2]
2020  GOSUB 6000
2025  FOR I=1 TO M2-1
2030    PRINT "WORKING!"
2035    FOR K=I+1 TO M2
2040      GOSUB 6100
2045      LET V=0
2050      GOSUB 3000
2055    NEXT K
2060  NEXT I
2065  OUTDVC "L2",E
2070  PRINT ES"U"
2075  PRINT
2080  PRINT
2085  GOSUB 5000
2090  PRINT TAB(20)"HPLC DATA ANALYSIS"
2095  PRINT TAB(20)"RUN ";S[2];DS[S[3]-3,S[3]];S[4]
2100  GOSUB 3300
2105  GOSUB 3500
2110  OUTDVC "T*",E
2115  PRINT
2120  PRINT "DO YOU WANT TO PLOT LINES OF RESOLUTION? (Y/N)";
2125  INPUT CS
2130  IF CS[1,1]# "Y" THEN STOP
2135  PRINT
2140  PRINT "WHAT IS THE MINIMUM RESOLUTION DESIRED ";
2145  INPUT R9
2150  PRINT "PEAK PAIRS ALWAYS RESOLVED GREATER THAN ";R9;"WILL BE IGNORED."
2155  OUTDVC "C1",E
2160  PRINT ES"U"
2165  LET Z1=2
2170  REM *** GET Y AND BETA VALUES ***
2175  GOSUB 6000
2180    FOR I=1 TO M2-1
2185      FOR K=I+1 TO M2

```

```

2190 GOSUB 6100
2195 PRINT
2200 PRINT
2205 PRINT
2210 REM *** CHECK FOR R9,-R9, AND 0 ***
2215 OUTDVC "T*",E
2220 PRINT ES"H",ES"J"
2225 PRINT " FOR PEAK PAIR ";I;"- ";K
2230 LET R7=Y=0
2235 PRINT "CHECKING FOR RESOLUTION OF ";Y
2240 GOSUB 5500
2245 IF R7=1 THEN 2295
2250 LET Y=R9
2255 PRINT "CHECKING FOR RESOLUTION OF ";Y
2260 GOSUB 5500
2265 IF R7=1 THEN 2295
2270 LET Y=-R9
2275 PRINT "CHECKING FOR RESOLUTION OF ";Y
2280 GOSUB 5500
2285 IF R7=1 THEN 2295
2290 IF Z1=3 THEN GOSUB 5700
2295 REM *** PRINT HEADING ***
2300 PRINT ES"b"
2305 OUTDVC "C1",E
2310 IF R7=1 THEN PRINT ES"U"
2315 PRINT
2320 PRINT
2325 PRINT " FOR PEAK PAIR ";I;"- ";K
2330 PRINT
2335 PRINT " BETA COEFFICIENTS (1-7)"
2340 PRINT D1;B2;B3;B4;B5;B6;B7
2345 PRINT
2350 PRINT
2355 REM *** CHECK POINTS ***
2360 PRINT " MEQH","ACN","THF","Y"
2365 PRINT "-----"
2370 LET X1=X2=.16
2375 LET X3=.68
2380 GOSUB 5800
2385 LET X1=X3=.16
2390 LET X2=.68
2395 GOSUB 5800
2400 LET X1=.68
2405 LET X2=X3=.16
2410 GOSUB 5800
2415 LET X1=X2=0
2420 LET X3=1
2425 GOSUB 5800
2430 LET X1=X3=0
2435 LET X2=1
2440 GOSUB 5800
2445 LET X1=1
2450 LET X2=X3=0
2455 GOSUB 5800
2460 PRINT "-----"
2465 PRINT
2470 PRINT
2475 IF R7=1 THEN LET Z1=0
2480 LET Z1=Z1+1
2485 IF R7=1 THEN GOSUB 4000

```

```

2490 IF R7=1 THEN PRINT ES"&p5D"ES"&p5u0C"
2495 IF R7#1 THEN PRINT "PEAK PAIR ";I;"- ";K;" IS ALWAYS RESOLVED ";R9
2500 IF R7=1 THEN GOSUB 5700
2505 NEXT K
2510 NEXT I
2515 OUTDVC "T*",E
2520 PRINT ES"b"
2525 PRINT "DO YOU WANT TO PLOT A DIFFERENT RESOLUTION (Y/N)";
2530 INPUT CS
2535 IF CS[1,1]="Y" THEN 2140
2540 STOP
3000 REM *** CALCULATES BEST SEPARATION SOLVENT ***
3005 READ #8,1
3010 READ #9,1
3015   FOR I1=0 TO 100 STEP 2
3020     FOR J1=0 TO 100-I1 STEP 2
3025       LET X1=I1/100
3030       LET X2=J1/100
3035       LET X3=1-X1-X2
3040       LET Y=B1*X1+B2*X2+B3*X3+B4*X1*X2+B5*X1*X3+B6*X2*X3+B7*X1*X2*X3
3045       READ #8,Y1
3050       IF ABS(Y) <= Y1 THEN LET Y1=ABS(Y)
3055       PRINT #9,Y1
3060       IF Y1 <= V THEN 3080
3065       LET V=Y1
3070       LET X6=I1
3075       LET X7=J1
3080     NEXT J1
3085   NEXT I1
3090 READ #8,1
3095 READ #9,1
3100   FOR I1=0 TO 100 STEP 2
3105     FOR J1=0 TO 100-I1 STEP 2
3110       READ #9,Y
3115       PRINT #8,Y
3120     NEXT J1
3125   NEXT I1
3130 RETURN
3300 REM *** PRINT BEST SEPARATION ***
3305 LET X1=X6/100
3310 LET X2=X7/100
3315 LET X3=1-X1-X2
3320 PRINT
3325 PRINT
3330 PRINT
3335 PRINT TAB(15);"THE BEST COMPOSITION IS:"
3340 PRINT
3345 PRINT TAB(10);"MEOH";TAB(25);"ACN";TAB(40);"THF"
3350 PRINT TAB(11);X6;"%";TAB(25);X7;"%";TAB(41);(100-X6-X7);"%"
3355 PRINT
3360 PRINT TAB(5);"THE WORST RESOLUTION AT THIS POINT IS ";V
3365 PRINT
3370 PRINT
3375 RETURN
3500 REM *** PREDICT ORDER OF ELUTION ***
3505 GOSUB 6000
3510 LET I=C[1]=1
3515 LET W=20
3520 LET B[1]=T[1,1]
3525   FOR K=2 TO M2

```

```

3530 LET C[K]=K
3535 GOSUB 6100
3540 LET Y=B1*X1+B2*X2+B3*X3+B4*X1*X2+B5*X1*X3+B6*X2*X3+B7*X1*X2*X3
3545 LET B[K]=W*Y+B[1]
3550 NEXT K
3555 REM *** SORT ***
3560 FOR I=1 TO M2-1
3565 FOR J=I+1 TO M2
3570 IF B[J] >= B[I] THEN 3605
3575 LET B1=B[I]
3580 LET C1=C[I]
3585 LET C[I]=C[J]
3590 LET C[J]=C1
3595 LET B[I]=B[J]
3600 LET B[J]=B1
3605 NEXT J
3610 NEXT I
3615 PRINT TAB(15); "PREDICTED ORDER OF ELUTION"
3620 PRINT
3625 PRINT TAB(10); "PEAK # ", "RELATIVE RETENTION TIME"
3630 PRINT
3635 FOR I=1 TO M2
3640 PRINT TAB(10); C[I]; TAB(35); B[I]/60
3645 NEXT I
3650 RETURN
4000 REM PLOT GRID
4001 OUTDVC "C1",E
4002 PRINT
4003 PRINT
4004 PRINT
4005 PRINT
4007 OUTDVC "T",E
4010 DIM I[21],J[21],L[21],M[21],N[21],O[21]
4030 LET G1=10
4040 LET G2=400
4050 LET K1=11
4060 LET D=G2/(K1-1)
4070 FOR I1=1 TO K1
4080 LET A=I1-1
4090 LET I[I1]=INT(G1+(0*G2+(1)*A*D)/1)
4100 LET J[I1]=INT(G1+(2*G2+(-1)*A*D)/2)
4110 LET L[I1]=INT(G1+(1*G2+(-1)*A*D)/2)
4120 LET M[I1]=INT(G1+(0*G2+(0)*A*D)*SQR(3)/2)
4130 LET N[I1]=INT(G1+(0*G2+(1)*A*D)*SQR(3)/2)
4140 LET O[I1]=INT(G1+(1*G2+(-1)*A*D)*SQR(3)/2)
4150 NEXT I1
4160 REM PRINT GRID
4170 PRINT ES"H"ES"J"ES"*m2a 0,0 jibz"ES"*dachlntz"
4180 LET M=K1-1
4190 FOR N=1 TO M
4200 LET L=N+1
4210 LET J=K1-N
4220 PRINT ES"*pah"I[L],M[L]"Z"ES"*pbh"L[J],O[J]"Z"
4230 PRINT ES"*pah"J[L],N[L]"Z"ES"*pbh"I[J],M[J]"Z"
4240 PRINT ES"*pah"L[L],O[L]"Z"ES"*pbh"J[J],N[J]"Z"
4250 NEXT N
4260 PRINT ES"*d0,0okSTHF"
4270 PRINT ES"*d422,0OACN"
4280 PRINT ES"*d230,3500MEOH"
4290 PRINT ES"*d1T"

```

```

4300 REM *** PLOT LINES OF RESOLUTION ***
4302 DIM X[101],Y[101],P[101],Q[101]
4303 LET Y=R9
4304 GOSUB 4310
4305 LET Y=-R9
4306 GOSUB 4310
4307 LET Y=0
4308 GOSUB 4310
4309 RETURN
4310 LET N=M+1
4311 FOR X=0 TO 100
4312 LET X1=X/100
4314 LET C1=B3+(B1-B3+B5-B5*X1)*X1
4316 LET C2=B2-B3+B6+(B4-B5-B6+B7-B7*X1)*X1
4318 LET C3=0-B6-B7*X1
4320 LET A=C3
4322 LET B=C2
4324 LET C=C1-Y
4326 LET D=B*B-4*A*C
4328 IF D<0 THEN 4370
4330 LET S=SQR(D)
4332 LET R1=(0-B+S)/(2*A)
4334 LET R2=(0-B-S)/(2*A)
4336 IF R1<0 THEN 4352
4338 IF R1>1 THEN 4352
4340 LET Q1=X1+R1
4342 IF Q1>1 THEN 4352
4344 LET X2=1-Q1
4346 LET X3=R1
4348 LET X[N]=G1+(X3+X1/2)*G2
4350 LET Y[N]=G1+X1*G2*SQR(3)/2
4351 LET N=N+1
4352 IF R2<0 THEN 4370
4354 IF R2>1 THEN 4370
4356 LET Q2=X1+R2
4358 IF Q2>1 THEN 4370
4360 LET X2=1-Q2
4362 LET X3=R2
4364 LET P[M]=G1+(X3+X1/2)*G2
4366 LET Q[M]=G1+X1*G2*SQR(3)/2
4368 LET M=M+1
4370 NEXT X
4372 IF N<3 THEN 4392
4374 LET Y8=Y[1]
4376 LET X8=X[1]
4378 FOR I9=2 TO N-1
4380 LET X9=X[I9]
4382 LET Y9=Y[I9]
4384 PRINT ES"*pah"x8,y8"Z"ES"*pbh"x9,y9"Z"
4386 LET X8=X9
4388 LET Y8=Y9
4390 NEXT I9
4391 PRINT ES"*dsZ";Y
4392 PRINT ES"*dtZ"
4393 IF M<3 THEN RETURN
4394 LET Y8=Q[1]
4396 LET X8=P[1]
4398 FOR I9=2 TO M-1
4400 LET X9=P[I9]
4402 LET Y9=Q[I9]

```

```

4404 PRINT ES"*pah"x8,y8"z"ES"*pbh"x9,y9"z"
4406 LET XP=X9
4408 LET YP=Y9
4410 NEXT I9
4412 PRINT ES"*dsz"
4413 PRINT Y
4414 PRINT ES"*dtz"
4416 RETURN
5000 REM *** GET DATE AND TIME ***
5010 DIM DS[48]
5020 LET DS="JAN FEB MAR APR MAY JUNEJULYAUG SEPTOCT NOV DEC "
5021 LET Z2=TIM(3)
5022 LET Z3=9
5023 LET Z4=12
5024 IF Z2/400=INT(Z2/400) LET M[2]=29
5025 IF Z2/100#INT(Z2/100) AND Z2/4=INT(Z2/4) LET M[2]=29
5026 LET Z2=TIM(2)
5027 FOR Z=1 TO 12
5028 LET Z2=Z2-M[Z]
5029 IF Z2 <= 0 THEN 5031
5030 NEXT Z
5031 LET S[2]=Z2+M[Z]
5040 LET S[4]=TIM(3)
5060 LET S[3]=Z*4
5070 RETURN
5500 REM *** CALCULATES PREDICTED RESOLUTION VALUES ***
5502 PRINT ES"c"
5504 FOR X=0 TO 100 STEP 2
5506 LET X1=X/100
5508 LET C1=B3+(B1-B3+B5-B5*X1)*X1
5510 LET C2=B2-B3+B6+(B4-B5-B6+B7-B7*X1)*X1
5512 LET C3=0-B6-B7*X1
5514 LET A=C3
5516 LET B=C2
5518 LET C=C1-Y
5520 LET D=B*B-4*A*C
5522 IF D<0 THEN 5548
5524 LET S=SQR(D)
5526 LET R1=(0-B+S)/(2*A)
5528 LET R2=(0-B-S)/(2*A)
5530 IF R1<0 OR R1>1 THEN 5538
5532 LET Q1=X1+R1
5534 IF Q1>1 THEN 5538
5536 LET R7=1
5538 IF R2<0 OR R2>1 THEN 5546
5540 LET Q2=X1+R2
5542 IF Q2>1 THEN 5546
5544 LET R7=1
5546 IF R7=1 THEN RETURN
5548 NEXT X
5550 RETURN
5700 REM *** NEW PAGE ***
5702 OUTDVC "C1",E
5704 PRINT ES"U"
5706 LET Z1=1
5708 RETURN
5800 REM *** CALCULATE PREDICTED RESOLUTION VALUES ***
5802 LET Y=B1*X1+B2*X2+B3*X3+B4*X1*X2+B5*X1*X3+B6*X2*X3+B7*X1*X2*X3
5804 PRINT X1*100,X2*100,X3*100,Y
5806 RETURN

```

```

6000 REM *** RESET POINTERS ***
6005 READ #1,1
6010 READ #2,1
6015 READ #3,1
6020 READ #4,1
6025 READ #5,1
6030 READ #6,1
6035 READ #7,1
6040 RETURN
6100 REM *** GET BETA COEFFICIENTS ***
6105 READ #1;Y1
6110 READ #2;Y2
6115 READ #3;Y3
6120 READ #4;Y4
6125 READ #5;Y5
6130 READ #6;Y6
6135 READ #7;Y7
6140 REM SOLVE FOR BETA COEFFICIENTS
6145 LET B1=Y1
6150 LET B2=Y2
6155 LET B3=Y3
6160 LET B4=4*Y4-2*(Y1+Y2)
6165 LET B5=4*Y5-2*(Y1+Y3)
6170 LET B6=4*Y6-2*(Y2+Y3)
6175 LET B7=27*Y7-12*(Y4+Y5+Y6)+3*(Y1+Y2+Y3)
6180 RETURN
9999 END

```

APPENDIX C

DATA REDUCTION FORTRAN 77 VERSION

This is basically a translated version of XLDB6A and B but with many enhancements. It still has a section to plot lines of resolution on a Hewlett-Packard graphics terminal with a graphics printer capable of a RASTER Dump but it is not limited to a graphics terminal. Extensive use of the Hewlett-Packard graphics package "DGL" has been made. This allows the user to use a 4 pen graphics plotter for output from a nongraphics terminal. Figure 1 of report text is typical of the output. This greatly simplifies selection of a solvent. Another enhancement is that the output gives expected retention time of named peaks for any selected composition and the minimum resolution predicted at that composition. Table 3 of report text is an example of this output.

```

1 FILES 2,2
2 SENS/TWO/,THREE/,FOUR/,SIX/
3 *****
4 *
5 *
6 *   INTEGER
7 *
8 *   NUMPEAK      # OF PEAKS CONSIDERED
9 *   LU           OUTPUT LU #
10 *  DATE         TODAYS DATE AND TIME
11 *  ORDER        THE PREDICTED ORDER OF ELUTION
12 *  PEN1PT       NUMBER OF POINTS FOR PEN 1
13 *  PEN2PT       NUMBER OF POINTS FOR PEN 2
14 *  PEN3PT       NUMBER OF POINTS FOR PEN 3
15 *  PEN4PT       NUMBER OF POINTS FOR PEN 4
16 *  ISAMPLE      INTEGER REPRESENTATION OF SAMPLE NAME
17 *  ICOLUMN      INTEGER REPRESENTATION OF COLUMN NAME
18 *  I_MEOH       INTEGER REPRESENTATION OF MEOH VALUE
19 *  I_ACN        INTEGER REPRESENTATION OF ACN VALUE
20 *  I_THF        INTEGER REPRESENTATION OF THF VALUE
21 *  I_SCALE      FRACTION OF RESOLUTION AT BEST COMP
22 *  PAGE         COUNTER FOR ITEMS ON A PAGE
23 *  PLOT         HOLDS DECISION TO PLOT LINES OF RESOL
24 *  XARRAY       ARRAY OF POINTS TO PLOT
25 *  YARRAY       ARRAY OF POINTS TO PLOT
26 *  PARRAY       ARRAY OF POINTS TO PLOT
27 *  QARRAY       ARRAY OF POINTS TO PLOT
28 *
29 *   REAL
30 *
31 *   COF          CHROMATOGRAPHIC OPTIMIZATION FUNCTION
32 *   VOIDRT       RETENTION TIME OF THE SOLVENT FRONT
33 *   WIDTH        PEAK WIDTH IN SECONDS
34 *   KPRIME       K' CAPACITY FACTOR
35 *   PEAKRT       PEAK RETENTION TIME IN MINUTES
36 *   RESWANT      RESOLUTION DESIRED
37 *   RESOLUTION   RESOLUTION BETWEEN PEAKS
38 *   MINKES       MINIMUM RESOLUTION FOR POINTS IN TRIANGLE
39 *   BESTRES      WORST RESOLUTION AT BEST CONDITIONS
40 *   TIME         PREDICTED RETENTION TIMES
41 *   BESTX1       AMOUNT OF MEOH AT BEST COMP
42 *   BESTX2       AMOUNT OF ACN AT BEST COMP
43 *   BESTX3       AMOUNT OF THF AT BEST COMT
44 *   I9,J9,L9,M9,N9,O9  ARRAYS OF TRIANGLE LINES
45 *   G1           OFFSET FOR PLOTTER
46 *   G2           MAXIMUM VALUE FOR PLOTTER
47 *   PEN1X        X COORDINATES FOR PEN 1
48 *   PEN1Y        Y COORDINATES FOR PEN 1
49 *   PEN2X        X COORDINATES FOR PEN 2
50 *   PEN2Y        Y COORDINATES FOR PEN 2
51 *   PEN3X        X COORDINATES FOR PEN 3
52 *   PEN3Y        Y COORDINATES FOR PEN 3
53 *   PEN4X        X COORDINATES FOR PEN 4
54 *   PEN4Y        Y COORDINATES FOR PEN 4
55 *   WIDE         MAXIMUM WINDOW WIDTH
56 *   HEIGHT       MAXIMUM WINDOW HEIGHT
57 *   VMAX         VIRTUAL MAXIMUM
58 *   VMIN         VIRTUAL MINIMUM
59 *   BETA1-BETA7  CHROMATOGRAPHIC BETA VALUES 1-7
60 *   Y            RESOLUTION AT A SPECIFIC COMPOSITION
61 *   FLOW         SOLVENT FLOW RATE (ml/min)

```

```

62 *      GO      RERUN FLAG      *
63 *      CHOSX1-CHOSX3      SELECTED SOLVENT COMPOSITION      *
64 *      SCALE      FRACTION OF THE BEST RESOLUTION      *
65 *      *      *      *
66 *      *      *      *
67 *      CHARACTER      *
68 *      *      *      *
69 *      PEAKNAME(35)*15      NAME OF THE PEAK      *
70 *      SOLV(7)*9      NAME OF SOLVENTS      *
71 *      QUEST*1      ANSWERS TO QUESTIONS      *
72 *      FF*1      FORM FEEL      *
73 *      ESC*1      ESCAPE CODE      *
74 *      FILE1*14      NAME OF DISC FILE HOLDING DATA      *
75 *      C_MEOH*4      CHARACTER REPRESENTATION OF MEOH VALUE      *
76 *      C_ACN*4      CHARACTER REPRESENTATION OF ACN VALUE      *
77 *      C_THF*4      CHARACTER REPRESENTATION OF THF VALUE      *
78 *      CSAMPLE*      CHARACTER REPRESENTATION OF SAMPLE NAME      *
79 *      CCOLUMN*16      CHARACTER REPRESENTATION OF COLUMN NAME      *
80 *      CSCALE*6      CHARACTER REPRESENTATION OF SCALE      *
81 *      *      *      *
82 *      *****      *
83 *      INITIALIZATION      *
84 *      *      *      *
85 *      PROGRAM LCMAN      *
86 *      COMMON /ONE/NUMPEAK,DATE,BESTX1,BESTX2,BESTX3,PEAKNAME      *
87 *      COMMON /TWO/BESTRES,MINRES,WIDTH,KPRIME,PEAKRT,VOIDRT      *
88 *      COMMON /THREE/RESOLUTION      *
89 *      COMMON /FOUR/PEN1X,PEN1Y,PEN2X,PEN2Y,PEN3X,PEN3Y,PEN4X,PEN4Y      *
90 *      COMMON /FIVE/PEN1PT,PEN2PT,PEN3PT,PEN4PT,ISAMPLE,ICOLUMN,      *
91 *      1 I_MEOH,I_THF,I_ACN,ISCALE      *
92 *      COMMON /SIX/XARRAY,YARRAY,PARRAY,QARRAY      *
93 *      COMMON ESC,FF,LU      *
94 *      INTEGER NUMPEAK,LU,DATE(15),ORDER(35),CONTRL,IERR,PEN1PT,      *
95 *      1 PEN2PT,PEN3PT,PEN4PT,ISAMPLE(8),ICOLUMN(8),I_MEOH(2),I_THF(2),      *
96 *      1 I_ACN(2),ISCALE(3),PAGE,PLOT,XARRAY(101),YARRAY(101),      *
97 *      1 PARRAY(101),QARRAY(101)      *
98 *      REAL COF(7),VOIDRT(7),WIDTH(7,35),KPRIME(7,35),PEAKRT(7,35),      *
99 *      1 RESWANT,RESOLUTION(7,35,35),MINRES(0:50,0:50),BESTRES,TIME(35),      *
100 *      1 BESTX1,BESTX2,BESTX3,XB(0:10),YB(0:10),XL(0:10),YL(0:10),      *
101 *      1 XR(0:10),YR(0:10),PEN1X(1300),PEN1Y(1300),PEN2X(1000),      *
102 *      1 PEN2Y(1000),PEN3X(1000),PEN3Y(1000),PEN4X(1000),PEN4Y(1000),      *
103 *      1 WIDE,HEIGHT,VMAX,VMIN      *
104 *      CHARACTER PEAKNAME(35)*15,SOLV(7)*9,FILE1*14,C_MEOH*4,C_ACN*4,      *
105 *      1 C_THF*4,CSAMPLE*16,CCOLUMN*16,CSCALE*6      *
106 *      CHARACTER *1 ESC,FF,QUEST      *
107 *      DIMENSION LUBUF(275)      *
108 *      EQUIVALENCE (C_MEOH,I_MEOH),(C_THF,I_THF),(C_ACN,I_ACN),      *
109 *      1 (CSAMPLE,ISAMPLE),(CCOLUMN,ICOLUMN),(CSCALE,ISCALE)      *
110 *      DATA SOLV(1),SOLV(2),SOLV(3)/'MEOH','ACN','THF'/      *
111 *      DATA SOLV(4),SOLV(5)/'MEOH&ACN','MEOH&THF'/      *
112 *      DATA SOLV(6),SOLV(7)/'ACN&THF','ALL THREE'/      *
113 *      ESC=CHAR(27)      *
114 *      FF=CHAR(12)      *
115 *      PEN1PT=0      *
116 *      PEN2PT=0      *
117 *      PEN3PT=0      *
118 *      PEN4PT=0      *
119 C      CHANGE THE FILE TO FIT YOUR SYSTEM !      *
120 *      FILE1(7)='::JC:3:9'      ! PREPARE FILE TO BE ON LU JC,TYPE 3      *
121 *      CALL LGBUF(LUBUF,275)      *
122 100 WRITE (1,290) ESC,ESC      ! CLEAR SCREEN AND PRINT QUESTION

```

```

123 READ (1,294) ICOLUMN
124 WRITE(1,(' ENTER THE SAMPLE NAME: _'))
125 READ (1,294) ISAMPLE
126 WRITE(1,(' DO YOU WANT TO USE AN OLD DATA FILE? _'))
127 READ (1,391) QUEST
128 IF (QUEST.NE.'Y') THEN
129   WRITE (1,291)
130   READ (1,293) FILE1(:6)
131   OPEN (333,FILE=FILE1,Iostat=IOS1,ERR=910,STATUS='NEW')
132 ELSE
133   WRITE (1,292)
134   READ (1,293) FILE1(:6)
135   OPEN (333,FILE=FILE1,Iostat=IOS1,ERR=910,STATUS='OLD')
136   REWIND (333)
137   READ (333) PEAKNAME
138   DO 105 I=1,7
139     READ (333) VOIDRT(I),(PEAKRT(I,J),WIDTH(I,J),J=1,35)
140 105 CONTINUE
141   WRITE (1,289)
142   READ *,NUMPEAK
143   IF (NUMPEAK.EQ.0) STOP
144   GOTO 225
145 ENDIF
146 *
147 **** GET PEAK DATA
148 *
149 200 WRITE (1,289)
150 READ *,NUMPEAK
151 IF (NUMPEAK.EQ.0) STOP
152 IF (NUMPEAK.LT.2.OR.NUMPEAK.GT.35) GOTO 200
153 DO 220 I=1,7
154   WRITE (1,280) SOLV(I)
155   READ *,VOIDRT(I)
156   IF (VOIDRT(I).EQ.0) STOP
157   IF (I.LT.2) THEN
158     WRITE (1,281)
159     DO 205 J=1,NUMPEAK
160       WRITE (1,282) J
161       READ *,PEAKRT(I,J),WIDTH(I,J),PEAKNAME(J)
162 205 CONTINUE
163   ELSE
164 210 WRITE (1,283)
165     DO 215 J=1,NUMPEAK
166       WRITE (1,284) PEAKNAME(J)
167       READ *,PEAKRT(I,J),WIDTH(I,J)
168 215 CONTINUE
169   ENDIF
170 220 CONTINUE
171 *
172 ***** PRINT INPUT
173 *
174 225 WRITE (1,285) VOIDRT
175 DO 230 I=1,NUMPEAK
176   WRITE (1,286) I,PEAKNAME(I),(PEAKRT(J,I),J=1,7)
177   WRITE (1,287) (WIDTH(J,I),J=1,7)
178 230 CONTINUE
179 WRITE (1,288)
180 280 FORMAT (/, 'ENTER THE RETENTION TIME (MIN) FOR SOLVENT ',A9,
181 1' <0=EXIT>')
182 281 FORMAT (/, 'ENTER THE RETENTION TIME (MIN), PEAK WIDTH (SEC),',
183 1' PEAK NAME',/, 'PEAK # RT WIDTH NAME')

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184 282 FORMAT (T2,I2,3X," ")
185 283 FORMAT (/, 'ENTER RETENTION TIME (MIN), AND PEAK WIDTH (SEC)',/,
186 1' PEAK RT WIDTH')
187 284 FORMAT (T2,A15,1X," ")
188 285 FORMAT (/,T48,'ACN&',T57,'THF&',T66,'ACN&',T74,'MEOH&',/,T21,
189 1'MEOH ACN THF MEOH MEOH THF ACN&THF',/,
190 1T22,'1',T31,'2',T40,'3',T49,'4',T58,'5',T67,'6',T76,'7',/,T19,
191 1'-----',/,
192 1T5,'SOLVENT FRONT',T21,F5.2,6F9.2,/)
193 286 FORIAT (I3,X1,A15,T21,F5.2,6F9.2)
194 287 FORMAT (T22,(' ',I3,' '),T31,(' ',I3,' '),T40,(' ',I3,' '),T49,(' ',I3,
195 1')',T58,(' ',I3,' '),T67,(' ',I3,' '),T76,(' ',I3,' '))
196 288 FORIAT (T19, '-----',
197 1'-----')
198 289 FORMAT ('ENTER THE NUMBER OF PEAKS PER SOLVENT. (2-35) <0=EXIT>')
199 290 FORMAT (A1,'H',A1,'J',/,T30,'HPLC OPTIMIZATION',/,
200 1' ENTER THE COLUMN NAME: ')
201 291 FORMAT (' ENTER THE NAME OF THE NEW FILE. ')
202 292 FORMAT (' ENTER THE NAME OF THE OLD FILE. ')
203 293 FORMAT (A6)
204 294 FORMAT (8A2)
205 295 FORIAT (A1,'H',A1,'J')
206 *
207 ***** CHANGE SECTION *****
208 *
209 300 WRITE (1,(' ARE THERE ANY CHANGES? _'))
210 READ (1,391) QUEST
211 IF (QUEST.NE.'Y') GOTO 320
212 WRITE (1,(' DO YOU WANT TO CHANGE A NAME? _'))
213 READ (1,391) QUEST
214 IF (QUEST.NE.'Y') GOTO 305
215 WRITE (1,(' ENTER THE PEAK # _'))
216 READ *,I
217 WRITE (1,394) I,PEAKNAME(I)
218 READ *,PEAKNAME(I)
219 GOTO 315
220 305 WRITE (1,(' WHICH SOLVENT? _'))
221 READ *,I
222 310 WRITE (1,('WHAT DO YOU WANT CHANGED? (VOIDRT,PEAKRT,OR WIDTH)'))
223 READ (1,391) QUEST
224 IF (QUEST.EQ.'V') THEN
225 WRITE (1,385) VOIDRT(I)
226 READ *,VOIDRT(I)
227 ELSEIF (QUEST.EQ.'P') THEN
228 WRITE (1,(' WHICH PEAK # ? _'))
229 READ *,J
230 WRITE (1,386) PEAKNAME(J),PEAKRT(I,J)
231 READ *,PEAKRT(I,J)
232 ELSEIF (QUEST.EQ.'W') THEN
233 WRITE (1,(' WHICH PEAK # ? _'))
234 READ *,J
235 WRITE (1,387) PEAKNAME(J),WIDTH(I,J)
236 READ *,WIDTH (I,J)
237 ELSE
238 GOTO310
239 ENDIF
240 WRITE (1,(' ANY OTHER CHANGES FOR THIS SOLVENT? _'))
241 READ (1,391) QUEST
242 IF (QUEST.EQ.'Y') GOTO 310
243 315 WRITE (1,('ANY OTHER CHANGES? _'))
244 READ (1,391) QUEST

```

```

245     IF (QUEST.EQ. 'Y') GOTO 305
246     GOTO 225
247 ***   WRITE THE DATA TO THE DISC FILE
248 320   REWIND (333)
249       WRITE (333) PEAKNAME
250       DO 325 I=1,7
251         WRITE (333) VOIDRT(I), (PEAKRT(I,J), WIDTH(I,J), J=1,35)
252 325   CONTINUE
253       CLOSE (333)
254       WRITE (1, ("DO YOU WANT TO ANALYSE THE ENTIRE LIST? <Y/N> _"))
255       READ (1,391) QUEST
256       IF (QUEST.EQ. 'Y') GOTO 360
257       WRITE (1, ("HOW MANY PEAKS DO YOU WANT TO ANALYSE? _"))
258       READ *, NUMPEAK
259       DO 335 I=1, NUMPEAK
260         WRITE (1, ("ENTER A PEAK NUMBER. _"))
261 330     READ *, ORDER(I)
262         WRITE (1,395) ORDER(I), PEAKNAME(ORDER(I))
263         READ (1,391) QUEST
264         IF (QUEST.NE. 'Y') THEN
265           WRITE (1, ("RE-ENTER THE PEAK #. _"))
266           GOTO 330
267         ENDIF
268 335   CONTINUE
269       DO 345 I=1, NUMPEAK-1
270         DO 340 J=I+1, NUMPEAK
271           IF (ORDER(J).LT. ORDER(I)) THEN
272             ORD1=ORDER(I)
273             ORDER(I)=ORDER(J)
274             ORDER(J)=ORD1
275           ENDIF
276 340   CONTINUE
277 345   CONTINUE
278       DO 355 I=1, NUMPEAK
279         PEAKNAME(I)=PEAKNAME(ORDER(I))
280         DO 350 J=1,7
281           PEAKRT(J,I)=PEAKRT(J, ORDER(I))
282           WIDTH(J,I)=WIDTH(J, ORDER(I))
283 350   CONTINUE
284 355   CONTINUE
285 360   DO 370 I=1,7
286         VRT=VOIDRT(I)*60
287         DO 365 J=1, NUMPEAK
288           RT=PEAKRT(I,J)*60
289           KPRIME(I,J)=(RT-VRT)/VRT
290 365   CONTINUE
291 370   CONTINUE
292 375   WRITE (1,388)
293       READ *, LU
294 C     CHANGE THE LU NUMBERS TO FIT YOUR SYSTEM
295       IF (LU.EQ.6) WRITE (LU,389) 1 FORM FEED LINE PRINTER
296       IF (LU.EQ.7.OR. LU.EQ.53) WRITE (LU,*) FF 1 FF 9871 OR 9876
297       IF (LU.NE.1.AND. LU.NE.6.AND. LU.NE.7.AND. LU.NE.53) GOTO 375
298       CALL FTIME( DATE)
299       WRITE (LU,390) 1 SKIP 3 LINES
300       WRITE (LU,392) DATE, C COLUMN, CSAMPLE
301       WRITE (LU,285) VOIDRT
302       DO 380 I=1, NUMPEAK
303         WRITE (LU,286) I, PEAKNAME(I), (PEAKRT(J,I), J=1,7)
304         WRITE (LU,287) (WIDTH(J,I), J=1,7)
305 380   CONTINUE

```

```

306 WRITE (LU,288)
307 385 FORMAT (" VOIDRT= ",F5.2," NEW VALUE IS TO BE ", " ")
308 386 FORMAT (T2,A15," RETENTION TIME = ",F7.2," NEW VALUE ", " ")
309 387 FORMAT (T2,A15," PEAK WIDTH = ",F7.2," NEW VALUE ", " ")
310 C CHANGE THE LU NUMBERS TO FIT YOUR SYSTEM
311 388 FORMAT (/, " WHERE DO YOU WANT THE OUTPUT ?",/,T10,"1 = THIS TERM",
312 1"INAL ",/,T10,"6 = THE LINE PRINTER",/,T10,"7 = THE DAISY WHEEL ",
313 1"PRINTER",/,T10,"53= THE THERMAL PRINTER")
314 389 FORMAT ("1")
315 390 FORMAT (/,/)
316 391 FORMAT (A1)
317 392 FORMAT (T30,"HPLC OPTIMIZATION",/,T23,15A2,/,T34,"INPUT DATA",/,
318 1T20,"COLUMN :",A16,T45,"SAMPLE :",A16,/,T25,"(DATA IS CORRECTED",
319 1" TO 1.00 ml/min)")
320 393 FORMAT (A15)
321 394 FORMAT (" PEAK ",I2," IS CURRENTLY ",A15,/, " ENTER THE NEW ",
322 1"NAME ")
323 395 FORMAT (7," PEAK # ",I2," IS CURRENTLY ",A15," OK? <Y/N> _")
324 *
325 ***** CALCULATE RESOLUTION *****
326 *
327 DO 410 I=1,7
328 DO 405 J=1,NUMPEAK-1
329 DO 400 K=J+1,NUMPEAK
330 RESOLUTION (I,J,K)=(PEAKRT(I,K)*60.-PEAKRT(I,J)*60.)/
331 ((WIDTH(I,K)+WIDTH(I,J))*0.5)
332 400 CONTINUE
333 405 CONTINUE
334 410 CONTINUE
335 WRITE (1,(" DO YOU WANT TO CALCULATE THE COF ? <Y/N> _"))
336 READ (1,391) QUEST
337 IF (QUEST.EQ. 'Y') THEN
338 415 WRITE (1,(" ENTER THE DESIRED RESOLUTION. _"))
339 READ *,RESWANT
340 IF (RESWANT.LT.0) GOTO 415
341 DO 430 I=1,7
342 COF(I)=0
343 DO 425 J=1,NUMPEAK-1
344 DO 420 K=J+1,NUMPEAK
345 Z=RESOLUTION(I,J,K)/RESWANT
346 IF (Z.LE.0) GOTO 420
347 COF(I)=COF(I)+ALOG(Z)
348 420 CONTINUE
349 425 CONTINUE
350 430 CONTINUE
351 *
352 *** OUTPUT
353 *
354 DO 450 I=1,7
355 C CHANGE THE LU NUMBERS TO FIT YOUR SYSTEM
356 IF (LU.EQ.6) WRITE (LU,389) 1 FORM FEED LINE PRINTER
357 IF (LU.EQ.7.OR.LU.EQ.53) WRITE (LU,*) FF 1 FF 9871 OR 9876
358 WRITE (LU,490) SOLV(I),VOIDRT(I)
359 DO 435 J=1,NUMPEAK
360 WRITE (LU,491) PEAKNAME(J),PEAKRT(I,J),WIDTH(I,J),
361 1 KPRIME(I,J)
362 435 CONTINUE
363 WRITE (LU,492)
364 DO 445 J=1,NUMPEAK-1
365 DO 440 K=J+1,NUMPEAK
366 I3=INDEX(PEAKNAME(J), " ")

```

```

367             IF (I3.GT.15.OR.I3.LT.1) I3=15
368             WRITE (LU,493) PEAKNAME(J)(:I3),PEAKNAME(K),
369             1      RESOLUTION(I,J,K)
370 440          CONTINUE
371 445          CONTINUE
372             WRITE (LU,494) SOLV(I),COF(I)
373 450          CONTINUE
374             ENDIF
375 490          FORMAT (///,T5,'TIME ZERO FOR SOLVENT ',A9,' IS',F7.2,
376 1' MIN. ',///, 'INGREDIENT',T20,' RETENTION TIME',T37,' BAND WIDTH',
377 1T50,' CAPACITY'./)
378 491          FORMAT (T2,A15,T25,F5.2,T40,I3,T51,F6.2)
379 492          FORMAT (///,T8,' FOR PEAKS',T35,' RESOLUTION',/)
380 493          FORMAT (T2,R15,' - ',A15,T36,F8.3)
381 494          FORMAT (///,' CHROMATOGRAPHIC OPTIMIZATION FUNCTION (COF) FOR ',
382 1A9,' IS',F9.4)
383 *
384 *****
385 *      CALLS TO SUBROUTINES
386 *
386 500          GO=0
387 505          CALL LCSORT (GO)
388             WRITE (1,('DO YOU WANT A PLOT OF THE RESOLUTION POINTS? _'))
389             READ (1,391) QUEST
390             IF (QUEST.EQ.'Y') CALL LCPOINT
391             WRITE (1,('DO YOU WANT A PLOT OF THE RESOLUTION LINES? _'))
392             READ (1,391) QUEST
393             IF (QUEST.EQ.'Y') CALL LCLINE
394             WRITE (1,('DO YOU WANT TO CALC FOR A DIFFERENT SOLVENT? _'))
395             READ (1,391) QUEST
396             GO=1
397             IF (QUEST.EQ.'Y') GOTO 505
398             WRITE (1,(' THE END !'))
399 C          CHANGE THE LU NUMBERS TO FIT YOUR SYSTEM
400             IF (LU.EQ.6) WRITE (LU,389) 1 FORM FEED LINE PRINTER
401             IF (LU.EQ.7.OR.LU.EQ.53) WRITE (LU,*) FF 1 FF 9071 OR 9076
402             STOP
403 *
404 ***** ERROR SECTION *****
405 *
406 910          WRITE (1,980) IOS1
407 980          FORMAT (///,'ERROR IN OPEN STATEMENT.  IOSTAT = ',I4)
408             END

```

```

409 *
410 ***** BEST SEPARATION SECTION *****
411 *
412 $EMA /TWO//THREE//FOUR//SIX/
413 SUBROUTINE LCSORT (GO)
414 COMMON /ONE/NUMPEAK,DATE,BESTX1,BESTX2,BESTX3,PEAKNAME
415 COMMON /TWO/BESTRES,MINRES,WIDTH,KPRIME,PEAKRT,VOIDRT
416 COMMON /THREE/RESOLUTION
417 COMMON /FIVE/PEN1PT,PEN2PT,PEN3PT,PEN4PT,ISAMPLE,ICOLUMN,
418 1 I_MEOH,I_THF,I_ACN,ISCALE
419 COMMON ESC,FF,LU
420 INTEGER NUMPEAK,LU,DATE(15),ORDER(35),CONTRL,IERR,PEN1PT,
421 1 PEN2PT,PEN3PT,PEN4PT,ISAMPLE(6),ICOLUMN(8),I_MEOH(2),I_THF(2),
422 1 I_ACN(2),ISCALE(3),PAGE,PLOT
423 REAL VOIDRT(7),WIDTH(7,35),KPRIME(7,35),PEAKRT(7,35),
424 1 RESOLUTION(7,35,35),MINRES(0:50,0:50),BESTRES,TIME(35),
425 1 BESTX1,BESTX2,BESTX3
426 CHARACTER PEAKNAME(35)*15,FILE1*14,C_MEOH*4,C_ACN*4,
427 1 C_THF*4,C_SAMPLE*16,CCOLUMN*16,CSCALE*6
428 CHARACTER *1 ESC,FF,QUEST
429 EQUIVALENCE (C_MEOH,I_MEOH),(C_THF,I_THF),(C_ACN,I_ACN),
430 1 (C_SAMPLE,ISAMPLE),(CCOLUMN,ICOLUMN),(CSCALE,ISCALE)
431 IF (GO.GT.0) GOTO 145
432 100 DO 110 I=0,50
433     DO 105 J=0,50-I
434         MINRES(I,J)=1.0E38
435 105 CONTINUE
436 110 CONTINUE
437 WRITE (1,388) ESC,ESC      ! REVERSE VIDIO AND PRINT "WORKING"
438 DO 130 I=1,NUMPEAK-1
439     IF (I.GT.1) WRITE (1,389) ESC,I,ESC ! REVERSE VIDIO AGAIN
440     DO 125 J=I+1,NUMPEAK
441         BETA1 = RESOLUTION (1,I,J)
442         BETA2 = RESOLUTION (2,I,J)
443         BETA3 = RESOLUTION (3,I,J)
444         BETA4 = 4.0*RESOLUTION (4,I,J) - 2.0*(BETA1+BETA2)
445         BETA5 = 4.0*RESOLUTION (5,I,J) - 2.0*(BETA1+BETA3)
446         BETA6 = 4.0*RESOLUTION (6,I,J) - 2.0*(BETA2+BETA3)
447         BETA7 = 27.0*RESOLUTION (7,I,J) - 12.0*(RESOLUTION (4,I,J) +
448 1 RESOLUTION (5,I,J) + RESOLUTION (6,I,J)) + 3.0*(BETA1+BETA2+BETA3)
449     DO 120 I1=0,100,2
450         DO 115 J1=0,100-I1,2
451             X1=I1/100.
452             X2=J1/100.
453             X3=1.0-X1-X2
454             Y=BETA1*X1+BETA2*X2+BETA3*X3+BETA4*X1*X2+BETA5*X1*X3+
455 1 BETA6*X2*X3+BETA7*X1*X2*X3
456             Y=ABS(Y)
457             I2=I1/2
458             J2=J1/2
459             IF (Y.LE.MINRES(I2,J2)) MINRES(I2,J2)=Y
460 115 CONTINUE
461 120 CONTINUE
462 125 CONTINUE
463 130 CONTINUE
464 *** SELECT THE BEST COMPOSITION AND ITS WORST RESOLUTION
465 WRITE (1,380) ESC,ESC      ! CLEAR THE SCREEN
466 BESTRES=0
467 DO 140 I=0,50
468     DO 135 J=0,50-I
469         IF (MINRES(I,J).GT.BESTRES) THEN

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```

470          BESTRES=MINRES(I,J)
471          BESTX1=I*2.0
472          BESTX2=J*2.0
473          BESTX3=100.-BESTX1-BESTX2
474          ENDIF
475 135      CONTINUE
476 140      CONTINUE
477 *
478 ***** PRINT BEST SEPARATION *****
479 *
480 C      CHANGE THE LU NUMBERS TO FIT YOUR SYSTEM
481 145  IF (LU.EQ.6) WRITE (LU,381)      I FORM FEED LINE PRINTER
482      IF (LU.EQ.7.OR.LU.EQ.53) WRITE (LU,*) FF I FF 9871 OR 9876
483      WRITE (LU,382)      I SKIP 3 LINES
484      WRITE (LU,385) DATE,CCOLUMN,CSAMPLE,BESTX1,BESTX2,BESTX3,BESTRES
485 *
486 ***** PREDICT ORDER OF ELUTION *****
487 *
488      CHOSX1=BESTX1
489      CHOSX2=BESTX2
490      CHOSX3=BESTX3
491      RES=BESTRES
492      WRITE (1,("WHAT FLOW RATE ARE YOU INTERESTED IN? _"))
493      READ *,FLOW
494      WRITE (1,("DO YOU WANT TO CHOSE YOUR OWN COMPOSITION? _"))
495      READ (1,383) QUEST
496      IF (QUEST.EQ.'Y') THEN
497      WRITE (1,385) DATE,CCOLUMN,CSAMPLE,BESTX1,BESTX2,BESTX3,BESTRES
498      WRITE (1,("ENTER COMPOSITION YOU WANT TO CALC RES TIMES FOR"))
499      READ *,CHOSX1,CHOSX2,CHOSX3
500      I=INT(CHOSX1/2.0)
501      J=INT(CHOSX2/2.0)
502      RES=MINRES(I,J)
503      IF (LU.EQ.6) WRITE (LU,381)      I FORM FEED LINE PRINTER
504      IF (LU.EQ.7.OR.LU.EQ.53) WRITE (LU,*) FF I FF 9871 OR 9876
505      WRITE (LU,384) DATE,CCOLUMN,CSAMPLE,CHOSX1,CHOSX2,CHOSX3,RES
506      ENDIF
507      DO 200 I=1,NUMPEAK
508          ORDER(I)=I
509 200  CONTINUE
510      DO 205 I=1,NUMPEAK
511          BETA1 = KPRIME(1,I)
512          BETA2 = KPRIME(2,I)
513          BETA3 = KPRIME(3,I)
514          BETA4 = 4.0*KPRIME(4,I)-2.0*(BETA1+BETA2)
515          BETA5 = 4.0*KPRIME(5,I)-2.0*(BETA1+BETA3)
516          BETA6 = 4.0*KPRIME(6,I)-2.0*(BETA2+BETA3)
517          BETA7 = 27.0*KPRIME(7,I)-12.0*(KPRIME(4,I)+KPRIME(5,I)+
518      1      KPRIME(6,I))+3.0*(BETA1+BETA2+BETA3)
519          X1=CHOSX1/100.
520          X2=CHOSX2/100.
521          X3=CHOSX3/100.
522          Y=(BETA1*X1+BETA2*X2+BETA3*X3+BETA4*X1*X2+BETA5*X1*X3+
523      1      BETA6*X2*X3+BETA7*X1*X2*X3)
524          TIME(I)=VOIDRT(1)*(1+Y)
525 205  CONTINUE
526 *
527 ***** SELECT ORDER *****
528 *
529      DO 215 I=1,NUMPEAK-1
530      DO 210 J=I+1,NUMPEAK

```

```

531         IF (TIME(J).LT.TIME(I)) THEN
532             ORD1=ORDER(I)
533             ORDER(I)=ORDER(J)
534             ORDER(J)=ORD1
535             T1=TIME(I)
536             TIME(I)=TIME(J)
537             TIME(J)=T1
538         ENDIF
539     210     CONTINUE
540     215     CONTINUE
541         WRITE (LU,386) FLOW
542         DO 220 I=1,NUMPEAK
543             J=ORDER(I)
544             WRITE (LU,387) I,PEAKNAME(J),TIME(I).TIME(I)/FLOW
545     220     CONTINUE
546 C     CHANGE THE LU NUMBERS TO FIT YOUR SYSTEM
547         IF (LU.EQ.6) WRITE (LU,381)          I FORM FEED LINE PRINTER
548         IF (LU.EQ.7.OR.LU.EQ.53) WRITE (LU,*) FF 1 FF 9871 OR 9876
549     380     FORMAT (A1,'H',A1,'J')
550     381     FORMAT ("1")
551     382     FORMAT (/,/)
552     383     FORMAT (A1)
553     384     FORMAT (T30,'HPLC OPTIMIZATION',/,T23,15A2,/,T32,
554         1'DATA ANALYSIS',/,T20,'COLUMN :',A16,T45,'SAMPLE :',A16,/,T25,
555         1'THE SELECTED COMPOSITION IS :',/,T21,'MEOH',T36,
556         1'ACN',T52,'THF',/,T21,I3,' ',T35,I3,' ',T51,I3,' ',/,T15,
557         1'THE WORST RESOLUTION AT THIS POINT IS ',F7.3,/)
558     385     FORMAT (T30,'HPLC OPTIMIZATION',/,T23,15A2,/,T32,
559         1'DATA ANALYSIS',/,T20,'COLUMN :',A16,T45,'SAMPLE :',A16,/,T25,
560         1'THE BEST COMPOSITION IS :',/,T21,'MEOH',T36,'ACN',T52,'THF',/,
561         1T21,I3,' ',T35,I3,' ',T51,I3,' ',/,T15,'THE WORST RESOLUTION',
562         1' AT THIS POINT IS ',F7.3,/)
563     386     FORMAT (/,T27,'PREDICTED ORDER OF ELUTION',/,T40,'RELATIVE ',
564         1'RETENTION TIME',/,T24,'INGREDIENT',T40,'1.00 ml/min ',
565         1F5.2,' ml/min',/)
566     387     FORMAT (T16,I2,T24,A15,T42,F6.2,T55,F6.2)
567     388     FORMAT (/,A1,'&dC WORKING! 1',A1,'&@')
568     389     FORMAT (A1,'&dC WORKING!',I3,A1,'&@')
569     RETURN
570     END

```

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571 *
572 ***** SUBROUTINE TO PLOT POINT GRAPH *****
573 *
574 $EMA /TWO/,/THREE/,/FOUR/,/SIX/
575 SUBROUTINE LCPOINT
576 COMMON /ONE/NUMPEAK,DATE,BESTX1,BESTX2,BESTX3,PEAKNAME
577 COMMON /TWO/BESTRES,MINRES,WIDTH,KPRIME,PEAKRT,VOIDRT
578 COMMON /FOUR/PEN1X,PEN1Y,PEN2X,PEN2Y,PEN3X,PEN3Y,PEN4X,PEN4Y
579 COMMON /FIVE/PEN1PT,PEN2PT,PEN3PT,PEN4PT,ISAMPLE,ICOLUMN,
580 1 I_MEOH,I_THF,I_ACN,ISCALE
581 COMMON ESC,FF,LU
582 INTEGER NUMPEAK,DATE(15),CONTRL,IERR,PEN1PT,
583 1 PEN2PT,PEN3PT,PEN4PT,ISAMPLE(8),ICOLUMN(8),I_MEOH(2),I_THF(2),
584 1 I_ACN(2),ISCALE(3),PAGE,PLOT
585 REAL WIDTH(7,35),KPRIME(7,35),PEAKRT(7,35),
586 1 RESWANT,MINRES(0:50,0:50),BESTRES,
587 1 BESTX1,BESTX2,BESTX3,XB(0:10),YB(0:10),XL(0:10),YL(0:10),
588 1 XR(0:10),YR(0:10),PEN1X(1300),PEN1Y(1300),PEN2X(1000),
589 1 PEN2Y(1000),PEN3X(1000),PEN3Y(1000),PEN4X(1000),PEN4Y(1000),
590 1 WIDE,HEIGHT,VMAX,VMIN
591 CHARACTER C_MEOH*4,C_ACN*4,
592 1 C_THF*4,C_SAMPLE*16,C_COLUMN*16,C_SCALE*6
593 CHARACTER *1 ESC,FF,QUEST
594 EQUIVALENCE (C_MEOH,I_MEOH),(C_THF,I_THF),(C_ACN,I_ACN),
595 1 (C_SAMPLE,ISAMPLE),(C_COLUMN,ICOLUMN),(C_SCALE,ISCALE)
596 *
597 *****
598 *
599 PLOT MINIMUM RESOLUTION
600
601 G1=20.
602 G2=350.
603 Y_INCR=G2*(SQRT(3.0)/2)/10.
604 DO 500 I1=0,10
605 1 I1=1
606 1 A3(I1)=INT(G1+(Y_INCR*I1)/(SQRT(3.0)/2))
607 1 YB(I1)=INT(G1)
608 1 XL(I1)=INT(G1+(Y_INCR*I1)/(SQRT(3.0)/1))
609 1 YL(I1)=INT(G1+(Y_INCR*I1))
610 1 XR(I1)=INT(G1+(Y_INCR*10+Y_INCR*I1)/(SQRT(3.0)/1))
611 1 YR(I1)=INT(G1+(Y_INCR*(10-I1)))
612 500 CONTINUE
613 WRITE(1,590) ESC,ESC 1 REVERSE VIDIO PRINT "PLOTING"
614 *
615 ***** SELECT PEN COLOR ***
616 *
617 SCALE=BESTRES/4.0
618 K=1
619 L=1
620 M=1
621 N=1
622 DO 510 I=0,50
623 1 DO 505 J=0,50-I
624 1 1 X1=I*2./100.
625 1 1 X2=J*2./100.
626 1 1 X3=1.0-X1-X2
627 1 1 X=G1+(1.0-X3)*G2/2+(1.0*X2*G2/2)
628 1 1 Y=G1+(X1*G2*SQRT(3.0)/2)
629 1 1 RES=MINRES(I,J)
630 1 1 IF (RES.LE.SCALE) THEN
631 1 1 1 PEN1X(K)=X
632 1 1 1 PEN1Y(K)=Y
633 1 1 1 PEN1PT=K

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```

632         K=K+1
633     ELSEIF (RES.LE.(SCALE*2.0)) THEN
634         PEN2X(L)=X
635         PEN2Y(L)=Y
636         PEN2PT=L
637         L=L+1
638     ELSEIF (RES.LE.(SCALE*3.0)) THEN
639         PEN3X(M)=X
640         PEN3Y(M)=Y
641         PEN3PT=M
642         M=M+1
643     ELSEIF (RES.LE.(SCALE*3.75)) THEN
644         PEN4X(N)=X
645         PEN4Y(N)=Y
646         PEN4PT=N
647         N=N+1
648     ENDIF
649 505     CONTINUE
650 510     CONTINUE
651 *
652 ***** PLOT TRIANGULAR GRAPH (USES H-P DGL GRAPHICS PACKAGE)
653 *
654     CONTRL=0
655     CALL ZBEGN
656 C     CHANGE "54" TO FIT YOUR SYSTEM
657     CALL ZDINT (54,CONTRL,IERR) ! INITIALIZE PLOTTER ON LU 54
658     VMIN=0.0
659     VMAX=1.0
660     WIDE =410 ! THE MAX X VALUE IN WORLD COORDINATES
661     HEIGHT=410 ! THE MAX Y VALUE IN WORLD COORDINATES
662     XLIM=225 ! THE MAX X DISTANCE IN MILLIMETERS
663     YLIM=225 ! THE MAX Y DISTANCE IN MILLIMETERS
664     CALL ZWIND (VMIN,WIDE,VMIN,HEIGHT)
665     CALL ZDLIM (0,XLIM,0,YLIM,IERR)
666     CALL ZASPK (XLIM,YLIM)
667     CALL ZVIEW (VMIN,VMAX,VMIN,VMAX)
668     LM1=10/2
669     DO 520 N1=0,5
670         CALL ZMOVE (XL(N1),YL(N1))
671         CALL ZDRAW (XB(N1),YB(N1))
672         CALL ZDRAW (XR(N1),YR(N1))
673         IF (N1.LT.LM1) THEN
674             N2=10-N1
675             CALL ZDRAW (XL(N2),YL(N2))
676             CALL ZDRAW (XB(N2),YB(N2))
677             CALL ZDRAW (XR(N2),YR(N2))
678         ENDIF
679         CALL ZDRAW (XL(N1),YL(N1))
680 520 CONTINUE
681     CALL ZMCUR
682     CALL ZCSIZ (8.75,12.5)
683     CALL ZCOLR (1)
684     X=5
685     Y=5
686     CALL ZMOVE (X,Y)
687     CALL ZTEXT (3,3HTHF)
688     X=360
689     Y=5
690     CALL ZMOVE (X,Y)
691     CALL ZTEXT (3,3HACN)
692     X=180

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693      Y=335
694      CALL ZMOVE (X,Y)
695      CALL ZTEXT (4,4HMeOH)
696      WRITE (C_MEOH,(I4)) BESTX1
697      WRITE (C_ACN,(I4)) BESTX2
698      WRITE (C_THF,(I4)) BESTX3
699      WRITE (C_SCALE,(F6.3)) SCALE
700      X1=30
701      X2=220
702      X3=240
703      X4=260
704      X5=280
705      X6=263
706      X7=300
707      X8=320
708      X9=310
709      X10=340
710      X11=360
711      X12=395
712      CALL ZCOLR(1)
713      CALL ZMOVE(X3,X11)
714      CALL ZTEXT(18,18H HPLC OPTIMIZATION)
715      CALL ZMOVE(X4,X10)
716      CALL ZTEXT(14,14H DATA ANALYSIS)
717      CALL ZMOVE(X3,X8)
718      CALL ZCSIZ(5.5,9.0)
719      CALL ZTEXT(30,DATE)
720      CALL ZMOVE(X4,X7)
721      CALL ZTEXT(9,9H COLUMN :)
722      CALL ZMOVE(X9,X7)
723      CALL ZTEXT(15,15H COLUMN)
724      CALL ZMOVE(X4,X5)
725      CALL ZTEXT(9,9H SAMPLE :)
726      CALL ZMOVE(X9,X5)
727      CALL ZTEXT(15,15H SAMPLE)
728      CALL ZMOVE(X4,X4)
729      CALL ZTEXT(26,26H THE BEST COMPOSITION IS )
730      CALL ZMOVE(X4,X3)
731      CALL ZTEXT(23,23H      MEOH      ACN      THF)
732      CALL ZMOVE(X5,X2)
733      CALL ZTEXT(4,4H MEOH)
734      CALL ZTEXT(1,1H%)
735      CALL ZMOVE(X8,X2)
736      CALL ZTEXT(4,4H ACN)
737      CALL ZTEXT(1,1H%)
738      CALL ZMOVE(X11,X2)
739      CALL ZTEXT(4,4H THF)
740      CALL ZTEXT(1,1H%)
741      *
742      ***** PLOT WORST RESOLUTION POINTS ***
743      *
744      CALL ZCOLR(1)
745      DO 535 I=1,PENIPT
746          WX=PENIX(I)
747          WY=PENIY(I)
748          CALL ZMOVE(WX,WY)
749          CALL ZMARK(8)
750      535 CONTINUE
751      CALL ZMOVE(X1,X11)
752      CALL ZMARK(8)
753      CALL ZTEXT(15,15H RESOLUTION < )

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754      CALL ZTEXT(6,ISCALE)
755      CALL ZMCUR
756      CALL ZCOLR(1)
757      WRITE(CSCALE,'(F6.3)') SCALE*2
758      DO 540 I=1,PEN2PT
759          WX=PEN2X(I)
760          WY=PEN2Y(I)
761          CALL ZMOVE(WX,WY)
762          CALL ZMARK(3)
763 540  CONTINUE
764      CALL ZMOVE(X1,X10)
765      CALL ZMARK(3)
766      CALL ZTEXT(15,15H RESOLUTION < )
767      CALL ZTEXT(6,ISCALE)
768      CALL ZMCUR
769      CALL ZCOLR(2)
770      WRITE(CSCALE,'(F6.3)') SCALE*3
771      DO 545 I=1,PEN3PT
772          WX=PEN3X(I)
773          WY=PEN3Y(I)
774          CALL ZMOVE(WX,WY)
775          CALL ZMARK(6)
776 545  CONTINUE
777      CALL ZMOVE(X1,X8)
778      CALL ZMARK(6)
779      CALL ZTEXT(15,15H RESOLUTION < )
780      CALL ZTEXT(6,ISCALE)
781      CALL ZMCUR
782      CALL ZCOLR(3)
783      WRITE(CSCALE,'(F6.3)') SCALE*3.75
784      DO 550 I=1,PEN4PT
785          WX=PEN4X(I)
786          WY=PEN4Y(I)
787          CALL ZMOVE(WX,WY)
788          CALL ZMARK(9)
789 550  CONTINUE
790      CALL ZMOVE(X1,X7)
791      CALL ZMARK(9)
792      CALL ZTEXT(15,15H RESOLUTION < )
793      CALL ZTEXT(6,ISCALE)
794      X=G1+(100-BESTX3)*G2/200+(BESTX2*G2/200)
795      Y=G1+(BESTX1*G2*SQRT(3.0)/200)
796      CALL ZCOLR(4)
797      CALL ZMOVE(X,Y)
798      CALL ZMARK(4)
799      CALL ZMARK(2)
800      WRITE(CSCALE,'(F6.3)') SCALE*4.0
801      CALL ZMOVE(X1,X5)
802      CALL ZMARK(4)
803      CALL ZMARK(2)
804      CALL ZTEXT(15,15H RESOLUTION = )
805      CALL ZTEXT(6,ISCALE)
806      CALL ZMOVE(X12,X6)
807      CALL ZMARK(4)
808      CALL ZMARK(2)
809      CALL ZMCUR
810      CALL ZDEND
811      CALL ZEND
812      WRITE(1,591) ESC,ESC,ESC ! CLEAR THE REVERSE VIDIO
813 590  FORMAT(A1,'&dc PLOTTING! PLOTTING! PLOTTING! ',A1,'&e')
814 591  FORMAT(A1,'A',A1,'J',A1,'B')
815      RETURN
816      END

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```

817 *
818 ***** SUBROUTINE TO PLOT LINES OF RESOLUTION *****
819 *
820 $EMA /TWO/,/THREE/,/FOUR/,/SIX/
821 SUBROUTINE LC LINE
822 COMMON /ONE/NUMPEAK,DATE,BESTX1,BESTX2,BESTX3,PEAKNAME
823 COMMON /THREE/RESOLUTION
824 COMMON /FIVE/PEN1PT,PEN2PT,PEN3PT,PEN4PT,ISAMPLE,ICOLUMN,
825 1 I MECH,I THF,I ACN,ISCALE
826 COMMON /SIX/XARRAY,YARRAY,PARRAY,QARRAY
827 COMMON ESC,FF,LU
828 INTEGER NUMPEAK,DATE(15),PEN1PT,PEN2PT,PEN3PT,PEN4PT,
829 1 ISAMPLE(8),ICOLUMN(8),I MECH(2),I THF(2),I ACN(2),ISCALE(3),
830 1 PAGE,PLOT,XARRAY(101),YARRAY(101),PARRAY(101),QARRAY(101)
831 REAL RESWANT,RESOLUTION(7,35,35),
832 1 XB(0:10),YB(0:10),XL(0:10),YL(0:10),XR(0:10),YR(0:10)
833 CHARACTER PEAKNAME(35)*15,CSAMPLE*16,CCOLUMN*16,CSCALE*6
834 CHARACTER *1 ESC,FF,QUEST
835 EQUIVALENCE (C MECH,I MECH),(C THF,I THF),(C ACN,I ACN),
836 1 (CSAMPLE,ISAMPLE),(CCOLUMN,ICOLUMN),(CSCALE,ISCALE)
837 *
838 *****
839 * PLOT LINES OF RESOLUTION
840 600 WRITE (1, "(" WHAT IS THE MINIMUM RESOLUTION DESIRED? ") ")
841 READ *,RESWANT
842 WRITE (1,870) RESWANT
843 PAGE=2
844 WRITE (1,887) ESC,ESC ! CLEAR THE SCREEN
845 WRITE (1,888) DATE,CCOLUMN,CSAMPLE,ESC,ESC !PRINT HEADING CN 9876
846 *
847 ***** GET BETA VALUES
848 *
849 DO 750 I=1,NUMPEAK-1
850 DO 745 J=I+1,NUMPEAK
851 WRITE (1,886) ESC ! TURN OFF GRAPHICS DISPLAY
852 BETA1 = RESOLUTION (1,I,J)
853 BETA2 = RESOLUTION (2,I,J)
854 BETA3 = RESOLUTION (3,I,J)
855 BETA4 = 4.0*RESOLUTION (4,I,J) - 2.0*(BETA1+BETA2)
856 BETA5 = 4.0*RESOLUTION (5,I,J) - 2.0*(BETA1+BETA3)
857 BETA6 = 4.0*RESOLUTION (6,I,J) - 2.0*(BETA2+BETA3)
858 BETA7 = 27.0*RESOLUTION (7,I,J) - 12.0*(RESOLUTION (4,I,J) +
859 1 RESOLUTION (5,I,J) + RESOLUTION (6,I,J)) + 3.0*(BETA1+BETA2+BETA3)
860 *
861 ***** CHECK FOR RESWANT, -RESWANT, AND 0
862 *
863 Y=0
864 PLOT=0
865 MARK=1
866 I3=INDEX(PEAKNAME(1), ' ')
867 IF (I3.GT.15.OR.I3.LT.1) I3=15
868 C CLEAR THE SCREEN AND PRINT THE PEAK PAIR
869 WRITE (1,871) ESC,ESC,PEAKNAME(I)(:I3),PEAKNAME(J)
870 WRITE (1,872) Y
871 GOTO 620
872 610 IF (PLOT.EQ.1) GOTO 640
873 Y=RESWANT
874 WRITE (1,872) Y
875 MARK=2
876 GOTO 620
877 615 IF (PLOT.EQ.1) GOTO 640

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```

878          Y=-RESWANT
879          WRITE (1,872)Y
880          MARK=3
881 *
882 ***** MAKES PLOT DECISION FOR A PEAK PAIR *****
883 *
884 620      DO 635 IX=0,100,2
885          X1=IX/100.
886          C1=BETA3+(BETA1-BETA3+3BETA5-BETA5*X1)*X1
887          C2=BETA2-BETA3+BETA6+(BETA4-BETA5-BETA6+BETA7-
888      1      BETA7*X1)*X1
889          C3=0-BETA6-BETA7*X1
890          A=C3
891          B=C2
892          C=C1-Y
893          D=B*B-4*A*C
894          IF (D.LT.0) GOTO 635
895          S=SQRT(D)
896          R1=(0-B+S)/(2*A)
897          R2=(0-B-S)/(2*A)
898          IF (R1.LT.0.OR.R1.GT.1) GOTO 625
899          Q1=X1+R1
900          IF (Q1.GT.1) GOTO 625
901          PLOT=1
902 625      IF (R2.LT.0.OR.R2.GT.1) GOTO 630
903          Q2=X1+R2
904          IF (Q2.GT.1) GOTO 630
905          PLOT=2
906 630      IF (PLOT.EQ.1) GOTO (610,615,640) MARK
907 631      CONTINUE
908          GOTO (610,615,640) MARK
909 640      IF (PAGE.GT.1.AND.PLOT.EQ.1.AND.J.GT.2) THEN
910          WRITE (1,873) ESC,ESC,ESC,ESC 1 FORM FEED FOR 9876
911          PAGE=1
912      ELSEIF (PAGE.GT.3) THEN
913          WRITE (1,873) ESC,ESC,ESC,ESC 1 FORM FEED FOR 9876
914          PAGE=1
915      ENDIF
916          WRITE (53,874) PEAKNAME(I)(:I3),PEAKNAME(J),
917      1      BETA1,BETA2,BETA3,BETA4,BETA5,BETA6,BETA7 1 OUTPUT TO 9876
918          WRITE (53,891) 1 OUTPUT TO 9876
919          X1=0.0
920          X2=0.0
921          X3=1.0
922          Y=BETA1*X1+BETA2*X2+BETA3*X3+BETA4*X1*X2+BETA5*X1*X3+
923      1      BETA6*X2*X3+BETA7*X1*X2*X3
924          WRITE (53,875)X1*100.,X2*100.,X3*100.,Y 1 OUTPUT TO 9876
925          X1=0.0
926          X2=1.0
927          X3=0.0
928          Y=BETA1*X1+BETA2*X2+BETA3*X3+BETA4*X1*X2+BETA5*X1*X3+
929      1      BETA6*X2*X3+BETA7*X1*X2*X3
930          WRITE (53,875)X1*100.,X2*100.,X3*100.,Y 1 OUTPUT TO 9876
931          X1=1.0
932          X2=0.0
933          X3=0.0
934          Y=BETA1*X1+BETA2*X2+BETA3*X3+BETA4*X1*X2+BETA5*X1*X3+
935      1      BETA6*X2*X3+BETA7*X1*X2*X3
936          WRITE (53,875)X1*100.,X2*100.,X3*100.,Y 1 OUTPUT TO 9876
937          WRITE (53,891) 1 OUTPUT TO 9876
938          PAGE=PAGE+1

```

```

939         IF (PLOT.NE.1) GOTO 740
940         WRITE (53,876)
941 ***      PLOT TRIANGLE ON CRT
942         G1=10.
943         G2=400.
944         Y_INCR=G2*(SQRT(3.0)/2)/10.
945         DO 645 I1=0,10
946             F=I1
947             XB(I1)=INT(G1+(Y_INCR*F)/(SQRT(3.0)/2))
948             YB(I1)=INT(G1)
949             XL(I1)=INT(G1+(Y_INCR*F)/(SQRT(3.0)/1))
950             YL(I1)=INT(G1+(Y_INCR*F))
951             XR(I1)=INT(G1+(Y_INCR*10+Y_INCR*F)/(SQRT(3.0)/1))
952             YR(I1)=INT(G1+(Y_INCR*(10-I1)))
953 645      CONTINUE
954 C        CLEAR SCREEN AND INITIALIZE 2648 GRAPHICS
955         WRITE (1,877) ESC,ESC,ESC,ESC
956         LM1=10/2
957         DO 650 N1=0,5
958             WRITE (1,878) ESC,XL(N1),YL(N1)
959             WRITE (1,880) ESC,XB(N1),YB(N1),ESC,XR(N1),YR(N1)
960             IF (N1.LT.LM1) THEN
961                 N2=10-N1
962                 WRITE (1,880) ESC,XL(N2),YL(N2),ESC,XB(N2),YB(N2),
963 1             ESC,XR(N2),YR(N2)
964             ENDIF
965             WRITE (1,880) ESC,XL(N1),YL(N1)
966 650      CONTINUE
967         WRITE (1,879) ESC,ESC,ESC,ESC ! LABEL THE TRIANGLE
968 *
969 ***** PLOT LINES OF RESOLUTION
970 *
971         Y=RESWANT
972         MARK=1
973         GOTO 700
974 655      Y=0
975         MARK=2
976         GOTO 700
977 660      Y=-RESWANT
978         MARK=3
979 ***** CALCULATES PREDICTED RESOLUTION VALUES
980 700      N=1
981         M=1
982         DO 715 X=0,100
983             X1=X/100
984             C1=BETA3+(BETA1-BETA3+BETA5-BETA5*X1)*X1
985             C2=BETA2-BETA3+BETA6+(BETA4-BETA5-BETA6+BETA7-
986 1             BETA7*X1)*X1
987             C3=0-BETA6-BETA7*X1
988             A=C3
989             B=C2
990             C=C1-Y
991             D=B*B-4*A*C
992             IF (D.LT.0) GOTO 715
993             S=SQRT(D)
994             R1=(0-B+S)/(2*A)
995             R2=(0-B-S)/(2*A)
996 705      IF (R1.LT.0.OR.R1.GT.1) GOTO 710
997             Q1=X1+R1
998             IF (Q1.GT.1) GOTO 710
999             X2=1-Q1

```

```

1000      X3=R1
1001      XARRAY(N)=G1+(X3+X1/2)*G2
1002      YARRAY(N)=G1+X1*G2*SQRT(3.0)/2
1003      N=N+1
1004 710    IF (R2.LT.0.OR.R2.GT.1) GOTO 715
1005      Q2=X1+R2
1006      IF (Q2.GT.1) GOTO 715
1007      X2=1-Q2
1008      X3=R2
1009      PARRAY(M)=G1+(X3+X1/2)*G2
1010      QARRAY(M)=G1+(X1*G2*SQRT(3.0)/2)
1011      M=M+1
1012 715    CONTINUE
1013      IF (N.LT.3) GOTO 725
1014      XMOVE=XARRAY(1)
1015      YMOVE=YARRAY(1)
1016 ***    DRAW RESOLUTION LINES ONTO TRIANGLE
1017      DO 720 I8=2,N-1
1018          XDRAW=XARRAY(I8)
1019          YDRAW=YARRAY(I8)
1020 C      DRAW RESOLUTION LINES
1021          WRITE (1,878) ESC,XMOVE,YMOVE
1022          WRITE (1,880) ESC,XDRAW,YDRAW
1023          XMOVE=XDRAW
1024          YMOVE=YDRAW
1025 720    CONTINUE
1026          WRITE (1,881) ESC,Y
1027 725    WRITE (1,882) ESC
1028          IF (M.LT.3) GOTO (655,660,735) MARK
1029          XMOVE=PARRAY(1)
1030          YMOVE=QARRAY(1)
1031          DO 730 I8=2,M-1
1032              XDRAW=PARRAY(I8)
1033              YDRAW=QARRAY(I8)
1034 C      DRAW RESOLUTION LINES
1035          WRITE (1,878) ESC,XMOVE,YMOVE
1036          WRITE (1,880) ESC,XDRAW,YDRAW
1037          XMOVE=XDRAW
1038          YMOVE=YDRAW
1039 730    CONTINUE
1040          WRITE (1,881) ESC,Y      I PRINT RESOLUTION ON TRIANGLE
1041          WRITE (1,882) ESC      I TURN OFF GRAPHICS TEXT
1042          GOTO (655,660,735) MARK
1043 735    WRITE (1,883) ESC,ESC I TRANSFER GRAPHICS TO 9876 VIA HP1B
1044          CALL WAIT (37,2)      I WAIT FOR RASTER DUMP TO FINISH
1045          WRITE (1,873) ESC,ESC,ESC I FORM FEED 9876
1046          PAGE=1
1047          IF (PLOT.EQ.1) GOTO 745
1048 740    WRITE (53,884) PEAKNAME(I)(:13),PEAKNAME(J),RESWANT
1049 745    CONTINUE
1050 750    CONTINUE
1051          WRITE (1,885)
1052          WRITE (1,886) ESC      I TURN OFF GRAPHICS DISPLAY
1053          READ (1,890) QUEST      I DUMMY TO TAKE "S" FROM PRINTER
1054          WRITE (1,889) ESC,ESC  I ERASE "S" FROM SCREEN
1055          READ (1,890) QUEST
1056          IF (QUEST.NE.'Y') RETURN
1057          GOTO 600
1058 870    FORMAT (///,'PEAK PAIRS ALWAYS RESOLVED GREATER THAN ',F5.2,
1059          1' WILL BE IGNORED!')
1060 871    FORMAT (A1,'H',A1,'J',///,'FOR PEAK PAIR ',A15,'- ',A15)

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1061 872 FORMAT (//, 'CHECKING FOR RESOLUTION OF ', F5.2)
1062 873 FORMAT (A1, 'Y', A1, 'U', A1, 'Z', A1, '&p3s5dB')
1063 874 FORMAT (//, T15, 'FOR PEAK PAIR ', A15, '-', A15, '//, T25, 'BETA ',
1064 1 'COEFFICIENTS (1-7) ', //, T10, 7F8.3, //, T23, 'MEOH', T37, 'ACN', T52,
1065 1 'THF', T65, 'RESOLUTION')
1066 875 FORMAT (T22, I4, ' ', T36, I4, ' ', T51, I4, ' ', T61, F12.3)
1067 876 FORMAT (//, //)
1068 877 FORMAT (A1, 'H', A1, 'J', A1, '*m2a 0,0 j1bZ', A1, '*dach1ntZ')
1069 878 FORMAT (A1, '*pah', 2I4, 'Z')
1070 879 FORMAT (A1, '*d 0,0 okSTHF', A1, '*d 400,0 OACN', A1, '*d 230,350 '
1071 1 'OleOH', A1, '*dlT')
1072 880 FORMAT (A1, '*obh', 2I4, 'Z')
1073 881 FORMAT (A1, '*dsZ', F5.2)
1074 882 FORMAT (A1, '*dtZ')
1075 883 FORMAT (A1, '*p5D', A1, '*p5u0C')
1076 884 FORMAT ('PEAK PAIR ', R15, '-', A15, ' IS ALWAYS RESOLVED ',
1077 1 'BETTER THAN ', F5.2, //)
1078 885 FORMAT (//, 'DO YOU WANT TO PLOT A DIFFERENT RESOLUTION? _')
1079 886 FORMAT (A1, '*dD')
1080 887 FORMAT (A1, 'H', A1, 'J')
1081 888 FORMAT (T30, 'HPLC OPTIMIZATION', //, T23, 15A2, //, T32,
1082 1 'DATA ANALYSIS', //, T20, 'COLUMN : ', A16, T45, 'SAMPLE : ', A16, //,
1083 1 T2, //, A1, 'H', A1, '*p3s5dF')
1084 889 FORMAT (A1, 'A', A1, 'J')
1085 890 FORMAT (A1)
1086 891 FORMAT (T17, '-----',
1087 1 '-----')
1088      END

```

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