AN7800

TO: W. C. HOLBROOK DATE: AUGUST 31, 1981

FROM: R. K. HINDERER

SUBJ: AUGUST 1981 PROGRESS REPORT

1. PVC/CPVC Pipe Task Force.

A steering committee (W. C. Becker, R. A. Krueger, K. Lee, M. M. O'Mara) for this task force has been formed. I have assisted L. Filer in developing a detailed proposal of objectives and activities for this task force.

2. IISRP.

BFG has terminated its membership in IISRP. After a discussion with K. Creene, I contacted IISRP to determine whether they would be receptive to my continued participation in selected areas. A response is expected shortly.

3. Cure-Rite 18 and OBTS.

The long-term feeding studies with Cure-Rite 18 and Santocure MOR (OBTS) continue to proceed satisfactorily.

4. Surpon.

We have initiated human patch testing with a Surpon absorbant polymer. Results are expected by the end of September. A test marketing program is planned if the results are favorable.

We have received a verbal report that Surpon extracts caused significant cell death in the cytotoxicity screen. This indicates that we will have to consider further studies to look at the effect of Surpon polymers on sensitive tissues. However, other verbal reports from one of our customers indicate that Surpon A-383 did not produce any detrimental effects in wound healing studies.

5. Combustion Toxicology.

Combustion toxicology studies with certain PVC, estane, and CPVC products are due to begin soon.

6. International/Europe.

We are continuing to work with International to develop a transition program to cope with unfinished projects that are affected by the sale of Ciago.

7. International/Australia.

We are continuing to work with Altona in an effort to obtain health related information for Australian food and drug approval of Bisphenol A.

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Am J Hosp Pharm. 1981; 38:1308–14

Interactions between Drugs and Polyvinyl Chloride Infusion Bags

Elizabeth A. Kowaluk, Michael S. Roberts, Harvey D. Blackburn, and Alan E. Polack

Forty-six injectable drug products, many of which are administered by i.v. infusion, were studied for loss from aqueous solutions stored in polyvinyl chloride infusion bags for various periods of time.

The polyvinyl bags were stored in the dark at room temperature for up to three months. Drugs stored in glass vials served as controls. The solutions were assayed spectrophotometrically at regular intervals. The effects of drug concentration and pH on the loss of drug from solution were studied. Octanol-water partition coefficients were used as a gauge of lipid solubility of the drugs.

Five of the drug products—clomethiazole edisylate, diazepam, hydralazine hydrochloride, thiopental sodium, and warfarin sodium—were found to be lost to a substantial extent after one week. For all drugs studied, the effect of the initial concentration on drug loss varied. The amount of drug lost over a given time was a function of the pH of the solution. The main physicochemical determinants controlling drug sorption appeared to be the extent of ionization and the lipid solubility of the drug.

For most of the drugs studied, minimal losses from the aqueous solutions were observed over short periods of storage time. Disappearance was slow and time dependent, indicating a diffusion-controlled sorption process. The losses of clomethiazole edisylate, thiopental sodium, and diazepam may be clinically important.

Index terms: Absorption; Anesthetics; Anticoagulants; Clomethiazole edisylate; Concentration; Containers; Diazepam; Diffusion; Hydralazine hydrochloride; Hydrogen ion concentration; Hypotensive agents; Injections; Ionization; Plastics; Polyvinyl chloride; Sedatives and hypnotics; Solubility; Stability; Storage; Thiopental sodium; Warfarin sodium

Previous studies[1–6] have reported the loss of certain drugs from aqueous solutions stored in plastic infusion bags for various periods of time. Generally, these losses have been attributed to interaction between the drug and the plastic infusion bag. Such interaction may result in reduced drug delivery to the patient and, in some cases, diminished therapeutic response.

Documentation of the compatibility of substances currently administered by intravenous administration sets, with the plastic infusion bags used in certain systems, is limited.[1–3,5–7] The most detailed evaluation of drug sorption by plastic infusion bags is in the study of Moorhatch and Chiou,[4] which reported on the sorption of 17 drugs. Although only limited information is available on the compatibility of drugs with polyvinyl chloride (the main resin in commercially available plastic infusion bags), the sorption of drugs by nylon and polyethylene has been evaluated extensively.[8–12] As the availability of medication through the intravenous route is of critical importance in patient therapy, it is essential to know not only which substances may be lost during intravenous infusion, but also which drugs may be safely administered using plastic intravenous delivery systems.

The present study was undertaken with the following intentions:

1. To survey a range of drugs, including those presently being administered by intravenous infusion, for possible interactions with plastic infusion bags,
2. To establish some of the physicochemical variables that control the rate, extent, and mechanism of such interactions to enable the subsequent development of criteria that may be used to predict their occurrence, and
3. To assess the possible clinical implications, if any, of those interactions that were found to occur.

The study consisted of several parts, the first being a preliminary survey of a range of drugs to identify the drugs that are lost from aqueous solution during storage in plastic infusion bags. The main goal of the survey was to evaluate the compatibility of medicaments routinely used in infusions by various Australian hospitals. The compatibility of antineoplastic medications with plastic infusion bags was not examined in this survey because of their restricted availability and cost.

In the second part of the study, the relative losses of some selected drugs in unbuffered aqueous solutions were compared with the losses in solutions buffered at pH 7.4. This study was undertaken because in situ pharmacologic studies are usually performed with drug solutions maintained at a pH of 7.4.[13]

Methods

Table 1 lists the drug products used in the preliminary survey. The drugs norepinephrine bitartrate[a] and isoproterenol hydrochloride[b] were examined; however, they were found to be unstable on storage and were not considered further.

Studies of the loss of all but two of the drugs from aqueous solution during storage in plastic infusion bags were conducted using 0.9% sodium chloride injection as the solvent. The study of the loss of sodium nitroprusside was performed with 5% dextrose injection as the solvent, and the study with clomethiazole edisylate used the infusion solution supplied by the manufacturer.

The preliminary survey was conducted using Viaflex (polyvinyl chloride) infusion bags[c] containing 500 ml of 0.9% sodium chloride injection or 500 ml of 5% dextrose injection in the case of sodium nitroprusside.

Each drug was prepared in an appropriate concentration so that when a practical volume (50 ml or less) was injected

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The donations of Travenol Laboratories and other pharmaceutical manufacturers in Australia are acknowledged. The assistance of the hospital pharmacy departments of Flinders Medical Center in Adelaide, Royal Hobart Hospital in Hobart, and Alfred Hospital in Melbourne, is acknowledged.

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drug was determined (Table 1). Blank solutions containing all components except the drug were used for adjustment of zero absorbance. In all cases, the drug solutions followed Beer's law over the concentration range studied. Samples of the following drug solutions were diluted immediately before assay: (1) acetazolamide sodium solutions were diluted 1:1 with phosphate buffer pH 8.1; (2) ampicillin trihydrate solutions were diluted 1:1 with phosphate buffer pH 7.4; (3) amoxicillin trihydrate and metronidazole solutions were diluted 1:1 with 0.2 *N* hydrochloric acid; (4) diazepam and dopamine hydrochloride solutions were diluted 1:1 with 0.2 *N* sulfuric acid; and (5) clomethiazole edisylate solutions were diluted 1:200 with distilled water.

The aminoglycoside antibiotics, gentamicin sulfate, kanamycin sulfate, and tobramycin sulfate, were assayed using the chemical derivatization procedure of Csiba.[14,15] This procedure involved the preparation of the dihydrolutidine derivative of the aminoglycoside and its subsequent spectrophotometric determination. In this study absorbance was measured at 336 nm rather than at 356 nm as suggested by Csiba.[15]

Samples of all other drug solutions were assayed undiluted.

The difference, if any, between the amount of drug in solution in the Viaflex bag and the amount in solution in the glass vial at any specific time was taken to be the amount of drug lost from solution during storage in the plastic infusion bag. Each result reported is the mean of the assays of at least two containers. The coefficient of variation for the assays was less than 10%.

Effect of pH on Loss. The effect of the pH of the drug solution in the plastic infusion bag on loss of drug from solution was studied for a number of selected drugs by buffering the solutions to a pH of 7.4 with a final phosphate buffer concentration of 0.02 *M*.

The experimental procedure was the same as the one used for the preliminary survey except that 50 ml of 0.9% sodium chloride injection from the Viaflex bags and 10 ml of 0.9% sodium chloride injection from the glass vials were replaced by an equivalent volume of a concentrated solution of the phosphate buffer (0.2 *M*). The initial concentrations were the same as in the preliminary survey.

Effect of Initial Concentration on Loss. The effect of varying the initial concentration of a drug solution on the loss of the drug was studied for a selection of drugs that showed losses of greater than 10% either from solutions buffered to pH 7.4 or from unbuffered solutions after storage for one week in plastic infusion bags.

These drugs were clomethiazole edisylate, hydralazine hydrochloride, thiopental sodium, and warfarin sodium in unbuffered solutions and chlorpromazine hydrochloride, promazine hydrochloride, and promethazine hydrochloride in buffered solutions.

Octanol-Water Partition Coefficients. Literature values for octanol-water partition coefficients have been used for all drugs studied where possible. When these values were not available for given compounds, partition coefficients were determined experimentally as follows. An aliquot of each drug solution was added to an aliquot of *n*-octanol[e] (analytical grade), which had been saturated with the particular diluent being used, in a glass bottle. (The proportions of aqueous phase to octanol phase were varied from 1:1 to 250:1 according to the affinity of a given drug for octanol.) Each bottle was sealed and agitated periodically during its storage in the dark at room temperature. Two aliquots of the drug solution were also placed into separate 50-ml glass bottles to act as controls. The aqueous phase was analyzed at weekly intervals until equilibrium was achieved. To prepare the solutions for assay, the content of the sample bottles was centrifuged at 3000 rpm for 10 minutes to achieve as complete as possible separation of aqueous and octanol phases. The octanol was aspirated off together with about 1 ml of the aqueous phase. The remainder of the aqueous phase was assayed spectrophotometrically (Table 1) and compared with the absorbance of the drug solutions from the control bottles.

The apparent octanol-water partition coefficients were determined for each drug according to the following equation:

$$K = [(A_{WI} - A_{WF})/A_{WF}]\,[V_W/V_O] \quad (1)$$

where K is the apparent octanol-water partition coefficient, A_{WI} is the initial absorbance of the aqueous drug solution, A_{WF} is the the final absorbance of the aqueous drug solution, V_W is the volume of the aqueous phase, and V_O is the volume of the octanol phase.

The apparent octanol-water partition coefficients for all of the drugs were adjusted for the pH of the individual infusion solutions using the following equation:

$$P_{infus} = P_{corr}\,(1 - \alpha_{infus}) = P_{app}\,(1 - \alpha_{infus})/(1 - \alpha_{app}) \quad (2)$$

where P_{infus} is the apparent octanol-water partition coefficient for a drug in a given infusion solution, P_{corr} is the true octanol-water partition coefficient (i.e., octanol-water partition coefficient of the unionized drug), P_{app} is the experimentally determined octanol-water partition coefficient, and α_{infus} and α_{app} are the degree of ionization of drug in the infusion solution and aqueous solution used for partition coefficient determination, respectively.

Results and Discussion

Preliminary Survey. Only five of the 46 drug products examined showed substantial loss after storage in plastic infusion bags for one week—clomethiazole edisylate, diazepam, hydralazine hydrochloride, thiopental sodium, and warfarin sodium (Table 1). Except for the phenothiazines, quinine sulfate, quinidine sulfate, and prednisolone, all drugs showing negligible loss from solution, had apparent octanol-water partition coefficients less than 5.

The majority of drugs appeared to be relatively stable in aqueous solutions stored in glass containers. After one week of storage, only methicillin sodium and cephalothin sodium showed any change in absorbance. In both cases an increase in absorbance was observed.

centration (Figure 4). During storage, the bags containing clomethiazole edisylate solutions seemed to become softer and more pliable, particularly at the higher concentrations. The distinctive odor of the solution was detectable in the immediate vicinity of the bags. It was apparent that clomethiazole edisylate altered the properties of the plastic and that this effect was a function of its concentration. Autian[8] reported a similar effect for the sorption of phenol by nylon and for the interaction of certain types of esters with rigid polyvinyl chloride bottles. He suggested that this occurred as a result of the tendency of these substances to act as solvents for the plastics,[8] in some instances causing greater polymer-chain flexibility through plasticization.[12] It is probable that clomethiazole acts in a similar manner. At high concentrations it "plasticizes" or "swells" the polyvinyl-chloride resin of the infusion bag. In this way, it enhances its own sorption and possibly its permeation through the plastic also. Fluck[9] and Roberts et al.[10] reported altered sorption and permeability characteristics for plastics in contact with some organic solvents, as well.

pH and Partition Coefficient. Figures 5 and 6 show plots of the percentage of thiopental sodium and promethazine hydrochloride, respectively, remaining in unbuffered and buffered (pH 7.4) aqueous solutions stored in plastic infusion bags for various periods of time. For the acidic drug thiopental sodium, sorption was fastest when the solution was unbuffered (pH 6.0) (Figure 5). At this pH, a greater proportion of the drug was unionized than at a pH of 7.4. In contrast, sorption of the basic drug promethazine was much greater at a pH of 7.4 when 1.96% of the drug was unionized, rather than at a pH of 5 when only 0.008% of the drug was present as the unionized form (Figure 6).

Table 2 shows that the percentage of other drugs lost from aqueous solutions stored in plastic infusion bags for one week was also a function of the solution pH. For a given drug, the extent of loss appeared to be directly related to the fraction unionized. In accordance with the classical pH-partition hypothesis, the greatest loss occurred for the drugs with the highest apparent octanol-water partition coefficients (Table 2). The correlation between percentage of drug lost during the storage of aqueous solutions in plastic infusion bags for one week and the apparent octanol-water partition coefficient (P_{app}) (Table 2) was poor ($r = 0.645$, $n = 23$). Therefore, equation 3, which describes that relationship, was of limited value in predicting the probable losses of other drugs.

$$\text{percentage loss} = 0.005\ P_{app} + 20.3 \qquad (3)$$

The poor correlation between the percentage loss and the apparent octanol-water partition coefficient can be partly ascribed to the inability of equation 3 to encompass the dynamic nature of the sorption process. For example, drugs with a high apparent octanol-water partition coefficient and a low percentage unionized (less than 0.1%) show a lower percent loss than predicted by equation 1; for those the percentage loss is governed by the rate of sorption of unionized drug.

Since the pH values of various infusion solutions available for clinical use vary,[16] it is possible to minimize the sorption of some lipophilic drugs by plastic infusion bags over short storage times by using infusion solutions with a pH such that the drugs are almost completely ionized in solution. Alternatively, a different i.v. delivery system[1] or storage in a refrigerator[17] could be used to minimize loss. Consideration of the pH of the solution in studies on drug-plastic interaction should be applied in clinical situations.

The extent of loss of drugs stored in plastic infusion bags may be enhanced by a reduction of the solution volume[5] or chemical instability.[12,18] These effects should be considered also in the use and storage of solutions in plastic infusion bags.

Conclusion

For most of the drugs studied, minimal losses of the drug from its aqueous solutions stored in plastic infusion bags over short time periods were observed. However, losses of clo-

Figure 5. Effect of pH of infusion solution on percentage of thiopental sodium remaining in aqueous solution during storage in plastic infusion bags: (●) buffered pH 7.4, (■) unbuffered pH ≃ 6.0.

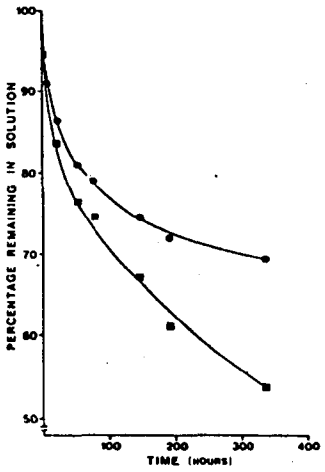
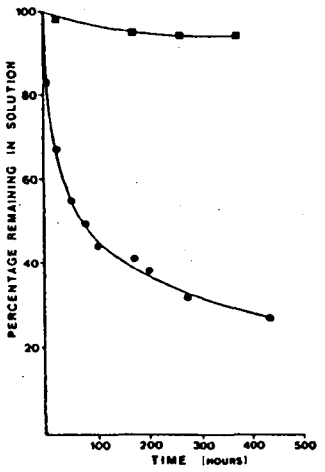


Figure 6. Effect of pH of infusion solution on percentage of promethazine hydrochloride remaining in aqueous solution during storage in plastic infusion bags: (●) buffered pH 7.4, (■) unbuffered pH ≃ 5.0.



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Am J Hosp Pharm. 1981; 38:1314–9

Stability of Five Catecholamines and Terbutaline Sulfate in 5% Dextrose Injection in the Absence and Presence of Aminophylline

David W. Newton, Esther Yin Yee Fung, and David A. Williams

The stability of dopamine hydrochloride, epinephrine hydrochloride, isoproterenol hydrochloride, methyldopate hydrochloride, norepinephrine bitartrate, and terbutaline sulfate in 5% dextrose injection with and without aminophylline was evaluated.

Kinetic data were obtained from the stability-indicating, high-performance liquid chromatographic assay method for autoxidation of the five sympathomimetic catecholamines and terbutaline sulfate.

The autoxidation of epinephrine hydrochloride, isoproterenol hydrochloride, norepinephrine bitartrate, and terbutaline sulfate was much faster in the alkaline solutions (pH 7.7–8.1) containing aminophylline than in the acidic solutions (pH 3.9–4.5) without aminophylline. On a molar basis among β_2 agonists, terbutaline sulfate was considerably more stable than epinephrine hydrochloride or isoproterenol hydrochloride. The autoxidation of terbutaline sulfate and the five catecholamines followed apparent zero-order kinetics. The rate constants and degradation times to fall to 90% of original potency were determined for each drug. Visual inspection was found to be grossly inadequate for estimating the stability of catecholamines to autoxidation.

Epinephrine hydrochloride, norepinephrine bitartrate, and isoproterenol hydrochloride should not be combined with aminophylline or similarly alkaline drugs in LVP solutions. The stability of very potent and life-saving drugs prepared for i.v. admixtures must be studied with stability-indicating assay methods.

Index terms: Additives; Aminophylline; Bronchodilators; Dextrose; Dopamine hydrochloride; Epinephrine hydrochloride; Hydrogen ion concentration; Incompatibilities; Injections; Isoproterenol hydrochloride; Kinetics; Methyldopate hydrochloride; Norepinephrine hydrochloride; Rate constants; Spasmolytics; Stability; Sympathomimetic agents; Terbutaline sulfate

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Address reprint requests to Dr. Newton, Department of Pharmaceutics, The University of Nebraska Medical Center, 42nd and Dewey Avenue, Omaha, NE 68105.

Donations of drug samples by American Critical Care, Ciba Pharmaceutical, Merck Sharp & Dohme, and Sterling-Winthrop are acknowledged. The assistance of Donna Earnshaw is acknowledged.

Abstracted in part from a thesis submitted to the graduate school, Massachusetts College of Pharmacy and Allied Health Sciences, in partial fulfillment of the requirements for the M.S. degree.

The autoxidation[a] of phenolic drugs, catecholamines, and related sympathomimetics is a common stability problem.[1–6] Several of these life-saving drugs, particularly the β_2 agonists epinephrine hydrochloride and isoproterenol hydrochloride, may be administered concurrently by intravenous infusion, possibly in i.v. admixtures, with aminophylline in clinical exigencies such as status asthmaticus and anaphylaxis.[7–10] The pharmacologic efficacy of these drugs is directly related to the stability of their phenolic hydroxy (Ar—OH)[b] groups.[4,11,12] An alkaline pH, such as that of aminophylline injection (8.6–9.0),[13] is a major determinant of the autoxidation of Ar—OH to form less sympathomimetically potent quinones (Ar=O).[1–6]

The usual agents that catalyze the oxidation of colorless phenolic drugs, often to colored products such as the semiquinones, quinones, and adrenochromes, were reviewed elsewhere.[14,15] The phenoxide anion ($Ar—O^-$) is more susceptible to oxidation than the undissociated Ar—OH group, and the oxidation rate of the —OH group decreases in the order of the *ortho*, *para*, and *meta* positions.[1–6,16]

The chemical stability of several sympathomimetic catecholamines mixed with large-volume parenteral (LVP) solutions has been reported.[15,17–21] These studies[15,17–21] found that catecholamine degradation (autoxidation) increases sharply above a pH of 6. Thus, substituting a typical pKa of 9 for the most acidic phenolic —OH group into equation 1,[14,22] it is apparent that as little as 0.1% of phenoxide ion can catalyze the oxidation of Ar—OH to Ar=O at a pH of 6.

$$\% \text{ dissociated} = 100/[1 + \text{antilog}(\text{pKa} - \text{pH})] \quad (1)$$

There appear to be no published data on the stability of catecholamines in i.v. admixtures with aminophylline, although warnings against preparing such solutions have been proffered.[13,23] The purpose of this investigation was to obtain kinetic data through a stability-indicating assay method for the autoxidation of five sympathomimetic catecholamines and terbutaline sulfate mixed with 5% dextrose injection in the presence and absence of aminophylline. Although currently terbutaline sulfate is only approved for subcutaneous injection, it was included because of its investigational i.v. use in treating premature labor.

Methods

HPLC Assay. A paired-ion high-performance liquid chromatographic (HPLC) assay using a variable wavelength fluorometric detector was developed because it offered the advantages of: (1) expediency, (2) specificity and sensitivity

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BFG TECHNICAL DOCUMENT

BFGoodrich
Chemical Division

RESEARCH AND DEVELOPMENT REPORT

[illegible] TESTING DATA ANALYSIS PROGRAM:
PART I - USERS' MANUAL

BY

D. T. POPOVICH

43824

Project No. 3572 **Report Type** Users' Manual

Profit Center Plastics **Date** Sept. 9, 1981

Copy Approval Authority D. E. Witenhafer [signature]

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BFG10709

PVC RESIN TESTING DATA ANALYSIS PROGRAMS PART I - USERS MANUAL

BY D T POPOVICH

ABSTRACT

Computer programs for the PVC resin testing data analysis have been written in a data file oriented fashion so as to provide a single summary report, including comments, of all tests run on a resin sample, while maintaining a record of multiple samples in progress These programs replace the Monroe calculator and are currently available for use in PVC manufacturing resin testing labs wherein computer support is available

SCOPE

For several years the data analysis in the PVC Resin Testing Lab was performed via the Monroe 1860 programmable calculator. These programs were for analysis of particle size and porosity (Hg) They were supplied to us, as to all production plants, by the manufacturing services group. In addition to these, we had written some additional programs to perform the calculations for Inherent Viscosity, Bulk Density etc. In anticipation of the obsolecence of the Monroe 1860, we decided to provide these same services as part of the installation of the ALTC Modcomp 7870. This was indeed one of the first tasks completed on this new computer.

Because the Modcomp 7870 is to serve as the host computer to several satellite computers which collect data from various experimental facilities, we decided to not only perform data analysis for the laboratory but to design the system such that it would be compatible with a more automated data retrieval system. In keeping with this goal, a system was designed to analyze data from the testing lab and maintain temporary files on each sample until testing is complete. Then a report of the test results is written. See the following page for an example of this summary report.

This users manual is Part I of the documentation for this system. Part II will deal with the mathematical treatment of the data and some programming considerations. Highly commented program listings are available from the author on request.

CONCLUSIONS AND SIGNIFICANCE

1 Programs for analyzing PVC Resin Testing data have been written in a high level programming language (FORTRAN IV).

2 These programs exist in two forms: (1) stand-alone versions which perform data analysis only and (2) a file oriented system which accumulates a summary report of all tests

3 These programs can be used in any of the production plants using Modcomp Computers with minimal changes and can easily be adapted for use on other computer equipment supporting FORTRAN IV

4 This system, which is a complete replacement for the Monroe calculator, has been written to easily interface with an ALTC data retrieval system to be developed in the future

PVC RESIN TESTING LAB — AVON LAKE TECH CENTER — BLDG 41J

SAMPLE IDENTIFICATION: MANUAL — PLANT LOCATION ALTC
REQUESTED BY POPOVICH — PROJECT NO 1234
DATE IN 9/ 4/1981 DATE OF REPORT 9/ 8/1981 TIME OF REPORT 10 12

P A R T I C L E S I Z E

MESH	% ON	CUMULATIVE % ON	CUMULATIVE % THRU
40	2.54	2.54	97.46
60	43.15	45.68	54.32
80	34.52	80.20	19.80
100	10.66	90.86	9.14
140	8.63	99.49	0.51
200	0.51	100.00	0.00
PAN	0.00	100.00	0.00

AVER PART SIZE = 241.37 MICRON
PART SIZE DIST = 33.33 PERCNT
COARSE FRACTION = 45.68 PERCNT
FINE FRACTION = 0.51 PERCNT

MESH	% ON	CUMULATIVE % ON	CUMULATIVE % THRU
40	0.00	0.50	99.50
60	1.51	2.01	97.99
80	22.11	24.12	75.88
100	29.15	53.27	46.73
140	34.67	87.94	12.06
200	9.05	96.99	3.01
PAN	3.01	100.00	-0.00

AVER PART SIZE = 153.06 MICRON
PART SIZE DIST = 33.42 PERCNT
COARSE FRACTION = 1.51 PERCNT
FINE FRACTION = 12.06 PERCNT

POROSITY (HG)	= 0.260	ML/GM
AVER PORE SIZE	= 0.672	MICRN
PORE SIZE DIST	= 99.558	%
FINES POROSITY	= 0.089	ML/G
FINE POR/TOTL POR	= 0.341	
INHERENT VISCOSITY	= 0.937	
APPARENT BULK DENS	= 0.509	GM/ML
COMPACT BULK DENS	= 0.583	GM/ML
FUNNEL FLOW	= 20.7	SEC
POWDER MIX TIME	= 350.0	SEC
DOP POROSITY	= 0.250	CC/GM
HEAT LOSS	= 0.090	%
BENNER FISH EYE	= 87	
STATE OF GLASS	= 1.730	
TOTAL SOLIDS	= 3.955	%
APP. PACK. FRAC	= 0.496	
COM. PACK. FRAC	= 0.568	

HERE WE CAN ADD ANY COMMENT IN FREE FORMAT UP TO 70 CHARACTERS
AND WE CAN CORRECT THEM BEFORE THEY ARE STORED
THIS IS A COMMENT ENTERED THROUGH THE COMMENT PROGRAM
WE CAN ALSO CORRECT THESE

SAMPLE ID MANUAL — REQUESTED BY POPOVICH DATE 9/ 8/1981

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LIST OF FIGURES

I INTRODUCTION

The QCLAB system is designed to accept data from the many tests performed in the resin testing lab, store these results in a file until all the tests from a particular sample have been completed and then write a comprehensive report of the results from these tests. In order to accomplish this, there are two types of programs involved in the system. The first category of programs is designed to control the files associated with each sample. The second category has programs that process test data and store the results in the files. Finally, two additional subjects will be addressed in this manual. These are the Terminal Monitor Program (TMP) and the Batch Processing Program (JOB).

II TERMINAL MONITOR PROGRAM (TMP)

The purpose of this program is to allow the user to control the activity at his terminal. There are three items related to TMP with which the QCLAB user need be concerned. These are: (1) the ability to start the BATCH program, (2) to abort an executing program or (3) to interrupt a program that is executing. To invoke TMP the user has the option of either of two actions: (1) issue a 'break' command with the break key on the terminal or (2) 'control A' where you must hold down the control key and the character 'A' simultaneously. In either of these cases TMP will be invoked and the computer will prompt the user with a '>' character to indicate that TMP is ready for a command. The user then will respond by:

1 RUN BAT

This will cause the BATCH (JOB) program to execute and the prompt character '$' to appear indicating that JOB is waiting for a command.

2 /A

This will cause the executing program to abort and return control to JOB

NOTE. It is good practice to issue a JOB command after aborting a program.

```
> e     $      (prompt character)
      JOB    (you input)
      $      (prompt character)
```

3 /R

This will cause an interrupted program to resume execution.

III BATCH PROCESSING PROGRAM (JOB)

The purpose of this program is to allow the execution of other programs and to establish the proper environment so that the input and output to these programs is properly directed. The prompt character for JOB is the '$' Whenever this character is the last item on the screen, JOB will accept a command. The key to the lab system is the JOB command 'QCLAB' followed by the name of the program that is to be executed. An optional third parameter may be entered in this sequence to allow output to the CRT rather than

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the Decwriter. This allows the suppression of the hard copy whenever it is not needed. A typical command to JOB would be

```
$                       (prompt character output by JOB)
QCLAB SAMPLE            (input by user to call the program SAMPLE)

or

$
QCLAB DIRECTORY TY      (input by user to execute the program
                         DIRECTORY and have the output appear
                         on the CRT rather than on the printer
```

IV. SAMPLE FILE ORIENTED PROGRAMS

IV.1 PROGRAM SAMPLE

This is the main program for sample file control. When the sample identification is entered, the program searches the files and attempts to locate the sample file. Sample identification may consist of one to sixteen characters entered in free form. Examples: SA 1234; SEG 4567;F76. Other examples can be found in figure 3.

During the search two possibilities exist:

1. A file by that name is not located and the program assumes a new file is going to be entered.

2. A file by that name is located and the program assumes an update of this file is required.

Each of these cases will be discussed.

New sample

If the sample is new, the program will ask for certain background information. This will include the name of the requester, the plant location and the project number. After this step is completed, the program will ask to have the tests which are to be run on this sample identified. A verification step is included in which the requested tests are output to the user for verification and correction. The tests which have been requested are then identified in the sample file. The requested tests are flagged in this manner so the system can track the tests which need to be run. The final task which is performed is to allow the entry of free-form comments about this sample. These may be used to identify tests which are to be run, but are not included in the list of standard tests. The results of these special tests may also be entered in the comment section of the files. However, these would be input using the program 'COMMENT' (see program 'COMMENT' below). A maximum of ten comments is allowed in each file.

Old sample

If the sample file which has been identified is already in the files, the program assumes that an update of this file is needed. First the program interrogates the user to determine if the file is to be deleted. If this is the case, a password must be entered before deletion occurs. If deletion is not intended, the program then proceeds to the 'ADD'/'DELETE' routine in which the requested tests are identified and corrections are made. The program will then go to the comment entry routine for additional comments. When this update is complete, the sample file is re-written. If this program is abnormally terminated (i.e. by an abort or some other system event), the file will not be re-written and the updates will not occur. In this case the file will remain as it was prior to this call.

Examples of these appear in Figure 1.

IV 2 PROGRAM COMMENT

This program allows the entry of comments into a sample file. The program asks for the sample identification. When the sample is located in the files the user enters the comment(s) in a free-form of up to 70 characters. Prior to writing the comment into the files the program will write it back to the user for verification. When the comment is verified it will be written into the file and entry of another comment will be requested. When all the comments have been entered, the command 'DONE' will terminate the program.

Example appears in Figure 2.

IV 3 PROGRAM DIRECTORY

The purpose of this program is twofold.

1. It lists all the samples currently in the file.

2. It serves as a reference to determine exactly how each sample is identified. If, when entering the sample identification into any of the programs in this system, any character or space is not exactly the same as when it was initialized, the sample will not be found.

Example appears in Figure 3.

IV 4 PROGRAM REPORT

This program is used to report the results. In addition, two other functions are performed by this program. The first of these is to check the data file and confirm that all data from all the tests that have been requested is present. If this is not the case, the tests for which data is missing will be identified and the user will be asked if an interim report should be printed. A final report will not be written until all the requested tests are reported. If certain tests are no longer needed, they must be

deleted using the program 'SAMPLE' before the final report will be allowed. Since the completion of the final report results in the deletion of the sample file, this routine provides some protection for the files. If all the data is present, a final report will be printed in triplicate. One of these copies will be identified to be kept in the lab as the permanent record of the test results. One copy will be identified to be sent to the requester of the tests. The third copy will be identified to be sent to data retrieval. When these three copies have been printed, the user will be asked to verify that they are complete. If the reports are complete, the sample is removed from the files. If the report is not satisfactory, the program will exit. After the cause of the unsatisfactory report is corrected, the user can recall this program and print out the report.

Interim report

This report can be issued if testing is not complete. Unlike the final report, only one copy will be printed. This report is also identified as an interim report. When this report is issued, the tests that have been requested and have not been reported will be flagged with a negative test value.

Examples of an interim and final report can be found in Figures 4 and 5.

V DATA ANALYSIS PROGRAMS

The purpose of each of the programs in this category is to accept data from the various tests, process that data and write the results of the data analysis or data input into the sample file.

V 1 COMMON CHARACTERISTICS

The programs in the data analysis category have certain common characteristics. These programs, like the file control programs, are called by the JOB command 'GCLAB XXXX'; where XXXX is the name of the program. In each of these programs the same method of sample file identification, sample file location and exiting is used. All the programs remain active until the exit is requested by the command 'DONE'. This allows many samples on which the same test has been performed to be processed without having to recall the program for each sample. In each of these programs, the user will be allowed two attempts to enter the sample identification. If the file is not located after the the second attempt, the program will exit to allow the user to check the file directory by means of program 'DIRECTORY' to verify the sample file exists and the correct identificaton is being used.

In the following sections a summary of the features of each program will be discussed. There are examples of each of these programs in the figures at the end of this manual. There are two types of programs in the data analysis category. Some of the programs in this category are merely vehicles for the data to be entered into the files. That is, some of the test results consist

of a single value and no calculations are necessary to evaluate the data. These are

1. BENNER FISH EYES (PROGRAM BEN)
2. FUNNEL FLOW (PROGRAM FF)
3. POWDER MIX TIME (PROGRAM PMT)

Each of the other programs in this series perform some type of calculation

V 2 STAND-ALONE PROGRAMS

Occasionally, a test may be required where the requester does not wish to have the entire report entered into the system. To accommodate this, each of the programs in which calculations are performed has a complimentary program that performs the calculations but does not interface to the sample files. These are called 'STAND-ALONE PROGRAMS'. These complimentary programs are called by appending an 'A' onto the regular name; for example 'APS' becomes 'APSA'. The following programs do data analysis and thus have stand-alone versions:

1. APPARENT BULK DENSITY. (PROGRAM ABD or ABDA)
2. PARTICLE SIZE ANALYSIS. (PROGRAM APS or APSA)
3. COMPACT BULK DENSITY. (PROGRAM CBD or CBDA)
4. DOP POROSITY. (PROGRAM DOP)
   no stand-alone version of this program
5. STATE OF GLASS. (PROGRAM GLAS or GLASA)
6. HEAT LOSS. (PROGRAM HL or HLA)
7. INHERENT VISCOSITY. (PROGRAM IV or IVA)
8. POROSITY (HG). (PROGRAM POR or PORA)
9. TOTAL SOLIDS. (PROGRAM TS or TSA)

V 3 PARTICLE SIZE

Program Features

1. There is currently a selection of four screen packs.

   a. Mesh sizes 40, 60, 80, 100, 140, 200
   b. Mesh sizes 60, 100, 170, 200, 230, 270
   c. Mesh sizes 20, 40, 50, 60, 80, 100
   d. Mesh sizes 40, 50, 60, 70, 80, 100

   One of these is selected prior to data entry

2. Because of the number of data points that are to be entered for this test, a data editing routine has been provided to allow the user to inspect and correct the data before any calculations are performed.

3. The data files have been set up to handle the results from two particle size analyses to accommodate those cases where one screen pack is not adequate. This feature is selected automatically. The program recognizes that one analysis is in

the file and indexes to the second storage area. The user should note that if a third set of results is entered, it will replace the second set. On those rare occasions when the user wishes to replace the first set of results, the results of the first test may be deleted using the program 'SAMPLE'. Then the particle size program will accept the next data set and enter the results into the first file position.

Example appears in Figure 6.

V 4 POROSITY (Mercury Intrusion)

There are two features of special note in the porosity analysis.

1. After the user has entered all the data for this program, an editing routine is called that allows the user to inspect the input data and correct the sample weight and/or the penetrometer "stem" readings. The program remains in this mode until the data is approved by the user.

2. Occasionally a request is made for the porosity of the fines from a sample and a report of the ratio of the fines porosity to the porosity from the entire sample. The porosity program has been written to accommodate this request. This is accomplished by having the program determine if a porosity result is already present in the file. If the result is there, as it must be for the fines porosity to be calculated, the user is given the option of entering the data as a fines porosity or replacing the results from the previous regular porosity. The selection of this option will be clear by inspecting the examples in Figure 7.

Example appears in Figure 7.

V 5 Apparent Bulk Density

There is just one feature of special note in this program. Normally, only one cup is used for this test in each lab. The tare weight of this cup is unlikely to change enough to affect the test. This tare weight has been put into the program and the user need not enter it. If an occasion should arise where this cup is changed, a change in the program will be required.

This program requires only the entry of the sample identification and the gross weight of the resin and the cup. Therefore no data correction features are included in this program. If an error is made in data entry and the results are written into the files, the user need only re-enter the sample identification and the correct gross weight. The results will be updated in the files.

Example appears in Figure 8.

V 6 Compact Bulk Density

This program is very much like the Apparent Bulk Density program One difference is that the tare weight of the graduate must be entered for each series of samples As a convenience to the user the tare weight of the graduate need only be entered once at the beginning of the program. Therefore, the program assumes that each sample was run with the same graduate. If for some reason the graduate was changed during a series of tests, the program must be recalled to enter the tare weight of the new graduate

In this program only two items of data are required for each sample (beside the graduate tare weight) These are the gross weight of the sample/graduate and the volume of the sample. This program does not provide any special data editing features. As with the other programs, if data correction is required, the user simple re-enters the sample identification and the corrected data and the files will be updated.

Example appears in Figure 9.

V. 7 POROSITY (DOP)

Program Features

1. The density of DOP at 30 deg. C. is part of this program. To accommodate the occasional use of another plasticizer or a change in density due to a change in temperature, the density value can be changed before the test data is entered.

Example appears in Figure 10.

The following programs have no special features and the operation of each of these is self explanatory.

STATE OF GLASS.....(GLAS)
HEAT LOSS.....(HL)
INHERENT VISCOSITY.....(IV)
TOTAL SOLIDS.....(TS)
BENNER FISH EYES.....(BEN)
FUNNEL FLOW.....(FF)
POWDER MIX TIME.....(PMT)

Examples of each of these programs appear in Figures 11 thru 17

FIGURE 1

```
OB
$
QCLAB SAMPLE
$ASS 1=TY 3=ATQ RAD=QCD LMT=QCM
$EXE SAMPLE.LMT
INPUT SAMPLE IDENTIFICATION (UP TO 16 CHARACTERS)
ENTER "DONE" AFTER LAST SAMPLE
MANUAL
ENTER EXPERIMENTER IDENTIFICATION (UP TO 8 CHAR.)
POPOVICH
ENTER LOCATION IDENTIFICATION (UP TO 8 CHAR.)
ALTC
ENTER PROJECT NUMBER
1234
ENTER TEST TO BE ADDED (ENTER DONE AFTER LAST TEST ENTERED)
?
THE TESTS NOW AVAILABLE ARE:
----------------------------

APS    AVERAGE PARTICLE SIZE  *    BEN   BENNER FISH EYE 6 MIN
APS2   SECOND SCREEN PACK     *    PMT   POWDER MIX TIME
POR    POROSITY               *    ABD   APPARENT BULK DENSITY
FPOR   FINES POROSITY         *    CBD   COMPACT BULK DENSITY
QLAS   STATE OF QLASS         *    FF    FUNNEL FLOW
DOP    DOP POROSITY           *    IV    INHERENT VISCOSITY
TS     TOTAL SOLIDS           *    HL    HEAT LOSS

PLEASE ENTER ONE OF THESE.

IF THE TEST YOU WISH DOES NOT APPEAR ON THIS LIST
THE RESULT MUST BE ENTERED THROUGH THE PROGRAM
"COMMENT" FOR IT TO APPEAR ON THE FINAL REPORT.
ENTER TEST TO BE ADDED (ENTER DONE AFTER LAST TEST ENTERED)
APS
ENTER TEST TO BE ADDED (ENTER DONE AFTER LAST TEST ENTERED)
APS2
ENTER TEST TO BE ADDED (ENTER DONE AFTER LAST TEST ENTERED)
POR
ENTER TEST TO BE ADDED (ENTER DONE AFTER LAST TEST ENTERED)
FPOR
ENTER TEST TO BE ADDED (ENTER DONE AFTER LAST TEST ENTERED)
QLAS
ENTER TEST TO BE ADDED (ENTER DONE AFTER LAST TEST ENTERED)
DOP
ENTER TEST TO BE ADDED (ENTER DONE AFTER LAST TEST ENTERED)
TS
ENTER TEST TO BE ADDED (ENTER DONE AFTER LAST TEST ENTERED)
BEN
ENTER TEST TO BE ADDED (ENTER DONE AFTER LAST TEST ENTERED)
PMT
ENTER TEST TO BE ADDED (ENTER DONE AFTER LAST TEST ENTERED)
ABD
ENTER TEST TO BE ADDED (ENTER DONE AFTER LAST TEST ENTERED)
CBD
```

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FIGURE 1 continued

```
ENTER TEST TO BE ADDED (ENTER DONE AFTER LAST TEST ENTERED)
FF
ENTER TEST TO BE ADDED (ENTER DONE AFTER LAST TEST ENTERED)
IV
ENTER TEST TO BE ADDED (ENTER DONE AFTER LAST TEST ENTERED)
HL
ENTER TEST TO BE ADDED (ENTER DONE AFTER LAST TEST ENTERED)
DONE
THE TESTS YOU HAVE REQUESTED ARE:
---------------------------------
APS
POR
ABD
CBD
FF
IV
HL
BEN
PHT
GLAS
APS2
DOP
TS
FPOR
IS THIS LIST COMPLETE AND CORRECT ?
NO      note: here is a example of how we update this file.
DO YOU WISH TO "ADD" OR "DELETE" A TEST
DELETE    note: here we will delete FF.
ENTER TEST TO DELETE (ENTER DONE AFTER LAST TEST IS ENTERED)
FF    note: this will remove FF from the list.
ENTER TEST TO DELETE (ENTER DONE AFTER LAST TEST IS ENTERED)
DONE    note: we are done deleting tests.
THE TESTS YOU HAVE REQUESTED ARE:
---------------------------------
APS
POR
ABD
CBD
IV
HL
BEN
PHT
GLAS
APS2
DOP
TS
FPOR
IS THIS LIST COMPLETE AND CORRECT ?
NO    note  let's put FF back in.
DO YOU WISH TO "ADD" OR "DELETE" A TEST
ADD
```

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FIGURE 1 continued

```
ENTER TEST TO BE ADDED (ENTER DONE AFTER LAST TEST ENTERED)
FF    note  FF goes back in.
ENTER TEST TO BE ADDED (ENTER DONE AFTER LAST TEST ENTERED)
DONE    note  we are now done adding.
THE TESTS YOU HAVE REQUESTED ARE
--------------------------------
APS
POR
ABD
CBD
FF
IV
HL
BEN
PMT
GLAS
APS2
DOP
TS
FPOR
IS THIS LIST COMPLETE AND CORRECT ?
YES    note: now we go on to the next step: COMMENTS
ARE THERE ANY ADDITIONAL TESTS THAT ARE TO BE RUN
OR OTHER COMMENTS YOU WISH TO MAKE ABOUT THIS SAMPLE ?
YES
ENTER COMMENT
HERE WE CAN ADD ANY COMMENT IN FREE FORMAT UP TO 70 CHARACTERS
HERE WE CAN ADD ANY COMMENT IN FREE FORMAT UP TO 70 CHARACTERS
IS THIS CORRECT ?
YES    note: the comment is verified as correct.
ANY MORE COMMENTS ?
YES    note: add another comment.
ENTER COMMENT
AND WE CAN COLLECT THEM BEFORE THEY ARE STORED
AND WE CAN COLLECT THEM BEFORE THEY ARE STORED
IS THIS CORRECT ?
NO
ENTER COMMENT
AND WE CAN CORRECT THEM BEFORE THEY ARE STORED
AND WE CAN CORRECT THEM BEFORE THEY ARE STORED
IS THIS CORRECT ?
YES ANY MORE COMMENTS ?
NO    note: we are now done and the program will exit.
```

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FIGURE 2

```
JOB
$
QCLAB COMMENT
$ASS 1=TY 3=ATQ RAD=QCD LMT=QCM
$EXE COMMENT.LMT



        * * * C O M M E N T * * *


ENTER SAMPLE IDENTIFICATION
MANUAL
ENTER COMMENT
THIS IS A COMMENT ENTERED THROUGH THE COMMENT PROGRAM
THIS IS A COMMENT ENTERED THROUGH THE COMMENT PROGRAM
IS THIS CORRECT ?
YES
ARE THERE ANY MORE COMMENTS YOU WOULD LIKE
TO ADD TO SAMPLE MANUAL
YES
ENTER COMMENT
WE CAN ALSO COLLECT THESE
WE CAN ALSO COLLECT THESE
IS THIS CORRECT ?
NO
ENTER COMMENT
WE CAN ALSO CORRECT THESE
WE CAN ALSO CORRECT THESE
IS THIS CORRECT ?
YES
ARE THERE ANY MORE COMMENTS YOU WOULD LIKE
TO ADD TO SAMPLE MANUAL
NO


            T H A N K   Y O U

            * * * B Y E * * *
```

FIGURE 3

```
JOB
$
QCLAB DIRECTORY
$ASS 1=TY 3=ATC RAD=QCD LMT=QCM
$EXE DIRECT.LMT


          * * * S A M P L E   D I R E C T O R Y * * *

SA 2139 25KPAS      9180C               SA 2148 755KPAS     9181C
F76                 WA 1504             9156                9164
SA 2145 81.86       SEQ 345             SA 2140 25KPAS      SEQ 346
SA 2143 755KPAS     9157                SEQ 347             SA 21461 60%
MANUAL              SA 2146 70%         WA 1504             SA 2136 25KPAS
SA 21461 755KPAS    9173                SEQ 348             9179
9175                9168                WA 1505             WA 1506


28 SAMPLES IN DIRECTORY

STOP DIREC


$JOB
$END QCLAB
$
```

FIGURE 4

```
JOB
$
QCLAB REPORT
$ASS 1=TY 3=ATG RAD=QCD LMT=QCM
$EXE REPORT.LMT



INPUT SAMPLE IDENTIFICATION
MANUAL
IS THIS A FINAL REPORT ?
NO    note  You will notice that this is an interim report
            The values of the test results which have been
            requested are all negative. We will output the
            final report in Figure 5.
```

PVC RESIN TESTING LAB AVON LAKE TECH CENTER BLDG 413

SAMPLE IDENTIFICATION MANUAL PLANT LOCATION ALTC
REQUESTED BY POPOVICH PROJECT NO 1234
DATE IN 9/ 4/1981 DATE OF REPORT 9/ 8/1981 TIME OF REPORT 8 49

PARTICLE SIZE

MESH	% ON	CUMULATIVE % ON	% THRU
-1	0.00	0.00	100.00
-1	0.00	0.00	100.00
-1	0.00	0.00	100.00
-1	0.00	0.00	100.00
-1	0.00	0.00	100.00
-1	0.00	0.00	100.00
PAN	0.00	0.00	100.00

AVER. PART. SIZE = -1.00 MICRON
PART. SIZE DIST. = -1.00 PERCNT
COARSE FRACTION. = 0.00 PERCNT
FINE FRACTION = 0.00 PERCNT

MESH	% ON	CUMULATIVE % ON	% THRU
-1	-0.00	0.00	100.00
-1	0.00	0.00	100.00
-1	0.00	0.00	100.00
-1	0.00	0.00	100.00
-1	0.00	0.00	100.00
-1	0.00	0.00	100.00
PAN	0.00	0.00	100.00

AVER. PART. SIZE = -1.00 MICRON
PART. SIZE DIST. = -1.00 PERCNT
COARSE FRACTION. = -0.00 PERCNT
FINE FRACTION = 0.00 PERCNT

POROSITY (HG)	= -1.000	ML/GM
AVER. PORE SIZE	= -1.000	MICRN
PORE SIZE DIST	= -1.000	%
FINES POROSITY	= -1.000	ML/GM
FINE POR/TOTL POR	= -1.000	
INHERENT VISCOSITY	= -1.000	
APPARENT BULK DENS	= -1.000	GM/ML
COMPACT BULK DENS	= -1.000	GM/ML
FUNNEL FLOW	= -1.0	SEC
POWDER MIX TIME	= -1.0	SEC
DOP POROSITY	= -1.000	CC/GM
HEAT LOSS	= -1.000	%
BENNER FISH EYE	= -1	
STATE OF GLASS	= -1.000	
TOTAL SOLIDS	= 0.000	%
APP. PACK. FRAC	= 0.000	
COM. PACK. FRAC	= 0.000	

HERE WE CAN ADD ANY COMMENT IN FREE FORMAT UP TO 70 CHARACTERS
AND WE CAN CORRECT THEM BEFORE THEY ARE STORED
THIS IS A COMMENT ENTERED THROUGH THE COMMENT PROGRAM
WE CAN ALSO CORRECT THESE

21134019

SAMPLE ID MANUAL REQUESTED BY: POPOVICH DATE 9/ 8/1981

INTERIM REPORT

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FIGURE 5

```
JOB
$
QCLAB REPORT
$ASS 1=TY 3=ATQ RAD=QCD LMT=QCM
$EXE REPORT.LMT



INPUT SAMPLE IDENTIFICATION
MANUAL
IS THIS A FINAL REPORT ?
YES    note: This is a final report. If any of the tests
             which have been requested have not been reported,
             the program will inform you at this time and
             prevent the final report from being written.



When the reports are written.


DID THE REPORT COME OUT ALL RIGHT ?
NO     note: This will cause the program to exit without
             removing the sample from the files.
             See section IV.4 Final Report.


YES    note: This will cause the file to be removed,
             and the program will then exit.
```

PVC RESIN TESTING LAB — AVON LAKE TECH CENTER — BLDG 413

SAMPLE IDENTIFICATION MANUAL — PLANT LOCATION ALTC
REQUESTED BY POPOVICH — PROJECT NO. 1234
DATE IN 9/ 4/1981 — DATE OF REPORT 9/ 8/1981 — TIME OF REPORT 10 12

PARTICLE SIZE

MESH	% ON	CUMULATIVE % ON	CUMULATIVE % THRU
40	2.54	2.54	97.46
60	43.15	45.68	54.32
80	34.52	80.20	19.80
100	10.66	90.86	9.14
140	8.63	99.49	0.51
200	0.51	100.00	0.00
PAN	0.00	100.00	0.00

AVER. PART. SIZE = 241.37 MICRON
PART. SIZE DIST. = 33.33 PERCNT
COARSE FRACTION. = 45.68 PERCNT
FINE FRACTION = 0.51 PERCNT

MESH	% ON	CUMULATIVE % ON	CUMULATIVE % THRU
40	0.00	0.50	99.50
60	1.51	2.01	97.99
80	22.11	24.12	75.88
100	29.15	53.27	46.73
140	34.67	87.94	12.06
200	9.05	96.99	3.01
PAN	3.01	100.00	-0.00

AVER. PART. SIZE = 153.06 MICRON
PART SIZE DIST. = 33.42 PERCNT
COARSE FRACTION. = 1.51 PERCNT
FINE FRACTION = 12.06 PERCNT

POROSITY (HG)	= 0.260	ML/GM
AVER. PORE SIZE	= 0.672	MICRN
PORE SIZE DIST	= 99.558	%
FINES POROSITY	= 0.089	ML/GM
FINE POR/TOTL POR	= 0.341	
INHERENT VISCOSITY	= 0.937	
APPARENT BULK DENS	= 0.509	GM/ML
COMPACT BULK DENS	= 0.583	GM/ML
FUNNEL FLOW	= 20.7	SEC
POWDER MIX TIME	= 350.0	SEC
DOP POROSITY	= 0.250	CC/GM
HEAT LOSS	= 0.090	%
BENNER FISH EYE	= 87	
STATE OF GLASS	= 1.730	
TOTAL SOLIDS	= 3.955	%
APP. PACK. FRAC	= 0.496	
COM. PACK. FRAC	= 0.568	

HERE WE CAN ADD ANY COMMENT IN FREE FORMAT UP TO 70 CHARACTERS
AND WE CAN CORRECT THEM BEFORE THEY ARE STORED
THIS IS A COMMENT ENTERED THROUGH THE COMMENT PROGRAM
WE CAN ALSO CORRECT THESE

21134021

SAMPLE ID. MANUAL — REQUESTED BY: POPOVICH DATE: 9/ 8/1981

COPY FOR LAB

FIGURE 6

```
JOB
$
QCLAB APS
$ASS 1=TY 3=ATQ RAD=QCD LMT=QCM
$EXE APS.LMT


* * * P A R T I C L E    S I Z E * * *


INPUT SAMPLE IDENTIFICATION (UP TO 16 CHARACTERS)
ENTER "DONE" AFTER LAST SAMPLE

MANUAL
          STANDARD SCREEN PACKS ARE:
                1          2          3          4
                -          -          -          -
               40         60         20         40
               60        100         40         50
               80        170         50         60
              100        200         60         70
              140        230         80         80
              200        270        100        100


INPUT DESIRED SCREEN PACK NUMBER
1    note: we are choosing screen pack 1.
INPUT TOTAL SAMPLE WEIGHT
2
INPUT SCREEN 1 GROSS AND TARE WEIGHTS
40.71 40.64
INPUT SCREEN 2 GROSS AND TARE WEIGHTS
38.09 37.24
INPUT SCREEN 3 GROSS AND TARE WEIGHTS
36.64 35.96
INPUT SCREEN 4 GROSS AND TARE WEIGHTS
35.44 35.23
INPUT SCREEN 5 GROSS AND TARE WEIGHTS
34.62 34.45
INPUT SCREEN 6 GROSS AND TARE WEIGHTS
33.63 33.92
INPUT PAN GROSS AND TARE WEIGHTS
155.28 155.28

          SCRN      GROSS       TARE
          ----      -----       ----
             1      40.71      40.64
             2      38.09      37.24
             3      36.64      35.96
             4      35.44      35.23
             5      34.62      34.45
             6      33.63      33.92
             7     155.28     155.28
```

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FIGURE 6 continued

```
DO YOU WISH TO CORRECT ANY OF THESE WEIGHTS ?
YES   note  tare on screen 6 is incorrect
ENTER SCREEN NUMBER TO BE CORRECTED
6
INPUT SCREEN 6 GROSS AND TARE WEIGHTS
33 63 33 62
          SCRN      GROSS       TARE
          ----      -----       ----
             1      40 71      40.66
             2      38.09      37.24
             3      36.64      35.96
             4      35.44      35.23
             5      34.62      34.45
             6      33 63      33.62
             7     155.28     155.28

DO YOU WISH TO CORRECT ANY OF THESE WEIGHTS ?
NO


* * * P A R T I C L E   S I Z E * * *


INPUT SAMPLE IDENTIFICATION (UP TO 16 CHARACTERS)
ENTER "DONE" AFTER LAST SAMPLE


DONE
STOP APS


$JOB
$END GCLAB
$
```

21134023

43824

FIGURE 6 continued

```
JOB
$
QCLAB APS
$ASS 1=TY 3=ATQ RAD=QCD LMT=QCM
$EXE APS.LMT


* * * P A R T I C L E   S I Z E * * *


INPUT SAMPLE IDENTIFICATION (UP TO 16 CHARACTERS)
ENTER "DONE" AFTER LAST SAMPLE

MANUAL
          STANDARD SCREEN PACKS ARE:
                1          2          3          4
                -          -          -          -
               40         60         20         40
               60        100         40         50
               80        170         50         60
              100        200         60         70
              140        230         80         80
              200        270        100        100


INPUT DESIRED SCREEN PACK NUMBER
1    note: we are choosing screen pack 1.
INPUT TOTAL SAMPLE WEIGHT
2
INPUT SCREEN 1 GROSS AND TARE WEIGHTS
40.67 40.64
INPUT SCREEN 2 GROSS AND TARE WEIGHTS
37.27 37.24
INPUT SCREEN 3 GROSS AND TARE WEIGHTS
36.40 35.96
INPUT SCREEN 4 GROSS AND TARE WEIGHTS
35.81 35.23
INPUT SCREEN 5 GROSS AND TARE WEIGHTS
35.14 34.48
INPUT SCREEN 6 GROSS AND TARE WEIGHTS
33.80 33.62
INPUT PAN GROSS AND TARE WEIGHTS
155.34 155.28

          SCRN      GROSS       TARE
          ----      -----       ----
             1      40.67      40.64
             2      37.27      37.24
             3      36.40      35.96
             4      35.81      35.23
             5      35.14      34.45
             6      33.80      33.62
             7     155.34     155.28
```

21134024

BFG10732

FIGURE 6 continued

```
DO YOU WISH TO CORRECT ANY OF THESE WEIGHTS
NO    note  no corrections are required.


* * * P A R T I C L E    S I Z E * * *


INPUT SAMPLE IDENTIFICATION (UP TO 16 CHARACTERS)
ENTER "DONE" AFTER LAST SAMPLE


DONE
STOP APS


$JOB
$END GCLAB
$
```

21134025

FIGURE 7

```
JOB
$
QCLAB POR
$ASS 1=TY 3=ATQ RAD=QCD LMT=QCM
$EXE POR.LMT


        P O R O S I T Y   P R O G R A M


INPUT SAMPLE IDENTIFICATION (UP TO 16 CHARACTERS)
ENTER "DONE" AFTER LAST SAMPLE
MANUAL


INPUT SAMPLE WEIGHT .1613
     6.5 PSIA STEM READING 0
     8.5 PSIA STEM READING .066
    10.5 PSIA STEM READING .072
    12.5 PSIA STEM READING .074
    14.4 PSIA STEM READING .076
    10.0 PSIG STEM READING .077
    20.0 PSIG STEM READING .079
    40.0 PSIG STEM READING .080
    60.0 PSIG STEM READING .082
    80.0 PSIG STEM READING .083
   100.0 PSIG STEM READING .084
   120.0 PSIG STEM READING .085
   140.0 PSIG STEM READING .086
   160.0 PSIG STEM READING .087
   180.0 PSIG STEM READING .089
   200.0 PSIG STEM READING .090
   300.0 PSIG STEM READING .095
   400.0 PSIG STEM READING .101
   500.0 PSIG STEM READING .107
  1000.0 PSIG STEM READING .119
  1500.0 PSIG STEM READING .121
  2000.0 PSIG STEM READING .122
  3000.0 PSIG STEM READING .124
  5000.0 PSIG STEM READING .124
```

21134026

FIGURE 7 continued

SAMPLE WEIGHT = 0 1613

PRESSURE	STEM	PRESSURE	STEM
6 5	0. 000	140. 0	0 086
8 5	0. 066	160. 0	0. 087
10 5	0. 072	180. 0	0 089
12. 5	0. 074	200. 0	0. 090
14 4	0. 076	300. 0	0. 095
10 0	0. 077	400. 0	0. 101
20 0	0. 079	500. 0	0. 107
40. 0	0. 080	1000. 0	0 119
60. 0	0. 082	1500. 0	0 121
80. 0	0. 083	2000. 0	0. 122
100. 0	0. 084	3000. 0	0. 124
120. 0	0. 085	5000. 0	0. 124

DO YOU WISH TO CORRECT ANY OF THESE VALUES YES
WEIGHT OR STEM ? WEIGHT
ENTER CORRECT WEIGHT . 1614

SAMPLE WEIGHT = 0. 1614

PRESSURE	STEM	PRESSURE	STEM
6. 5	0. 000	140. 0	0. 086
8. 5	0. 066	160. 0	0. 087
10. 5	0. 072	180. 0	0. 089
12. 5	0. 074	200. 0	0. 090
14. 4	0. 076	300. 0	0. 095
10. 0	0. 077	400. 0	0. 101
20. 0	0. 079	500. 0	0. 107
40. 0	0. 080	1000. 0	0. 119
60. 0	0. 082	1500. 0	0. 121
80. 0	0. 083	2000. 0	0. 122
100. 0	0. 084	3000. 0	0. 124
120. 0	0. 085	5000. 0	0. 124

DO YOU WISH TO CORRECT ANY OF THESE VALUES YES
WEIGHT OR STEM ? STEM

FIGURE 7 continued

```
ENTER PRESSURE OF STEM READING TO BE CORRECTED 2000
ENTER CORRECT STEM READING  121


          SAMPLE WEIGHT = 0.1614

    PRESSURE       STEM        PRESSURE       STEM
    --------       ----        --------       ----

         6.5      0.000           140.0      0.086
         8.5      0.066           160.0      0.087
        10.5      0.072           180.0      0.089
        12.5      0.074           200.0      0.090
        14.4      0.076           300.0      0.095
        10.0      0.077           400.0      0.101
        20.0      0.079           500.0      0.107
        40.0      0.080          1000.0      0.119
        60.0      0.082          1500.0      0.121
        80.0      0.083          2000.0      0.121
       100.0      0.084          3000.0      0.124
       120.0      0.085          5000.0      0.124
DO YOU WISH TO CORRECT ANY OF THESE VALUES NO


          P O R O S I T Y   P R O G R A M


INPUT SAMPLE IDENTIFICATION (UP TO 16 CHARACTERS)
ENTER "DONE" AFTER LAST SAMPLE
DONE
STOP POR


$JOB
$END @CLAB
$
```

21134028

FIGURE 7 continued

```
$B
$
$CLAB POR
$ASS 1=TY 3=ATG RAD=QCD LMT=QCM
$EXE POR.LMT


        P O R O S I T Y   P R O G R A M


INPUT SAMPLE IDENTIFICATION (UP TO 16 CHARACTERS)
ENTER "DONE" AFTER LAST SAMPLE
MANUAL
THE POROSITY FOR MANUAL              HAS BEEN ENTERED
IS THIS A FINES POROSITY ?
YES


INPUT SAMPLE WEIGHT  1563
     6.5 PSIA STEM READING 0
     8.5 PSIA STEM READING .002
    10.5 PSIA STEM READING .010
    12.5 PSIA STEM READING .010
    14.4 PSIA STEM READING .012
    10.0 PSIG STEM READING .013
    20.0 PSIG STEM READING .014
    40.0 PSIG STEM READING .015
    60.0 PSIG STEM READING .016
    80.0 PSIG STEM READING .016
   100.0 PSIG STEM READING .016
   120.0 PSIG STEM READING .016
   140.0 PSIG STEM READING .016
   160.0 PSIG STEM READING .017
   180.0 PSIG STEM READING .017
   200.0 PSIG STEM READING .017
   300.0 PSIG STEM READING .019
   400.0 PSIG STEM READING .022
   500.0 PSIG STEM READING .024
  1000.0 PSIG STEM READING .027
  1500.0 PSIG STEM READING .028
  2000.0 PSIG STEM READING .029
  3000.0 PSIG STEM READING .030
  5000.0 PSIG STEM READING .031
```

21134029

FIGURE 7 continued

SAMPLE WEIGHT = 0 1563

PRESSURE	STEM	PRESSURE	STEM
6.5	0.000	140.0	0.016
8.5	0.002	160.0	0 017
10.5	0.010	180.0	0.017
12.5	0.010	200.0	0.017
14.4	0.012	300.0	0.019
10.0	0.013	400.0	0.022
20.0	0.014	500.0	0.024
40.0	0.015	1000.0	0.027
60.0	0.016	1500.0	0.028
80.0	0.016	2000.0	0.029
100.0	0.016	3000.0	0.030
120.0	0.016	5000.0	0.031

DO YOU WISH TO CORRECT ANY OF THESE VALUES NO

P O R O S I T Y P R O G R A M

INPUT SAMPLE IDENTIFICATION (UP TO 16 CHARACTERS)
ENTER "DONE" AFTER LAST SAMPLE
DONE
STOP POR

$JOB
$END $CLAS
$

2113030

FIGURE B

```
JOB
$
GCLAB ABD
$ASS 1=TY 3=ATG RAD=GCD LMT=GCM
$EXE ABD, LMT



        * * *  APPARENT BULK DENSITY  * * *

INPUT SAMPLE IDENTIFICATION (UP TO 16 CHARACTERS)
ENTER "DONE" AFTER LAST SAMPLE
MANUAL
INPUT GROSS WEIGHT
275 55    note: The net weight of the cup is stored in the
                program. See section V.9.
INPUT SAMPLE IDENTIFICATION (UP TO 16 CHARACTERS)
ENTER "DONE" AFTER LAST SAMPLE
DONE
STOP ABD


$JOB
$END GCLAB
$
```

FIGURE 9

```
 08
$
$LAB CBD
$ASS 1=TY 3=ATG RAD=QCD LMT=QCM
$EXE CBD.LMT



        * * *  COMPACT BULK DENSITY  * * *

ENTER THE WEIGHT OF THE GRADUATE
187.6
INPUT SAMPLE IDENTIFICATION (UP TO 16 CHARACTERS)
ENTER "DONE" AFTER LAST SAMPLE
MANUAL
INPUT GROSS WEIGHT AND VOLUME OF SAMPLE
312.42 214
INPUT SAMPLE IDENTIFICATION (UP TO 16 CHARACTERS)
ENTER "DONE" AFTER LAST SAMPLE
DONE
STOP CBD


$JOB
$END QCLAB
$
```

21134032

FIGURE 10

```
JOB
$
GCLAB DOP
$ASS 1=TY 3=ATG RAD=GCD LMT=GCM
$EXE DOP.LMT



        * * *   D O P    P O R O S I T Y   * * *

I AM USING THE DENSITY FOR P-75 PLASTICIZER AT
30 DEG  C  WHICH IS  977 GM/CC
DO YOU WISH TO CHANGE THIS VALUE ?
NO
INPUT SAMPLE IDENTIFICATION (UP TO 16 CHARACTERS)
ENTER "DONE" AFTER LAST SAMPLE
MANUAL
ENTER TARE WEIGHT OF TUBE
27.4
ENTER GROSS WEIGHT BEFORE PLASTICIZER
32.4
ENTER GROSS WEIGHT AFTER PLASTICIZER
33.62
INPUT SAMPLE IDENTIFICATION (UP TO 16 CHARACTERS)
ENTER "DONE" AFTER LAST SAMPLE
DONE
STOP DOP


$JOB
$END GCLAB
$
```

FIGURE 11

```
JOB
$
QCLAB GLAS
$ASS 1=TY 3=ATQ RAD=QCD LMT=QCM
$EXE GLAS.LMT



        * * *  S T A T E  O F  G L A S S  * * *

INPUT SAMPLE IDENTIFICATION (UP TO 16 CHARACTERS)
ENTER "DONE" AFTER LAST SAMPLE
MANUAL
INPUT THE SAMPLE WEIGHT, COUNTER READING AND TURNS

15 9.5 1.5 INPUT SAMPLE IDENTIFICATION (UP TO 16 CHARACTERS)
ENTER "DONE" AFTER LAST SAMPLE
DONE
STOP GLAS


$JOB
$END QCLAB
$
```

FIGURE 12

```
JOB
$
GCLAB HL
$ASS 1=TY 3=ATQ RAD=QCD LMT=QCM
$EXE HL.LMT



        * * *  H E A T   L O S S  * * *

INPUT SAMPLE IDENTIFICATION (UP TO 16 CHARACTERS)
ENTER "DONE" AFTER LAST SAMPLE
MANUAL
INPUT WET, DRY, AND PAN WEIGHTS
6.2979 6.2935 1.4087
INPUT SAMPLE IDENTIFICATION (UP TO 16 CHARACTERS)
ENTER "DONE" AFTER LAST SAMPLE
DONE
STOP HL


$JOB
$END GCLAB
$
```

FIGURE 13

```
JOB
●
QCLAB IV
$ASS 1=TY 3=ATQ RAD=QCD LMT=QCM
$EXE IV.LMT



*** INHERENT VISCOSITY ***

ENTER BLANK DROP TIMES (2)
226.8 226.7

ENTER SAMPLE I.D. ("DONE" AFTER LAST SAMPLE)
MANUAL
SAMPLE WEIGHT, DROP TIMES (2)
 2017 273.8 274.0

ENTER SAMPLE I.D. ("DONE" AFTER LAST SAMPLE)
DONE
STOP IV


$JOB
$END QCLAB
$
```

FIGURE 14

```
JOB
$
GCLAB TS
$ASS 1=TY 3=ATG RAD=GCD LMT=GCM
$EXE TS.LMT



INPUT SAMPLE IDENTIFICATION (UP TO 16 CHARACTERS)
ENTER "DONE" AFTER LAST SAMPLE
MANUAL
INPUT WET, DRY AND PAN WEIGHTS FOR PAN 1
4.9430 1.5655 1.4265
INPUT WET, DRY AND PAN WEIGHTS FOR PAN 2
4.8475 1.5633 1.4280
INPUT SAMPLE IDENTIFICATION (UP TO 16 CHARACTERS)
ENTER "DONE" AFTER LAST SAMPLE
DONE
STOP TS


$JOB
$END GCLAB
$
```

FIGURE 15

```
JOB
$
GCLAB BEN
$ASS 1=TY 3=ATG RAD=GCD LMT=GCM
$EXE BEN.LMT



        * *  B E N N E R  F I S H  E Y E S  * *

INPUT SAMPLE IDENTIFICATION (UP TO 16 CHARACTERS)
ENTER "DONE" AFTER LAST SAMPLE
MANUAL
INPUT BENNER FISH EYE COUNT FOR MANUAL
87
INPUT SAMPLE IDENTIFICATION (UP TO 16 CHARACTERS)
ENTER "DONE" AFTER LAST SAMPLE
DONE
STOP BEN


$JOB
$END GCLAB
$
```

2134038

FIGURE 16

```
JOB
$
QCLAB FF
$ASS 1=TY 3=ATG RAD=QCD LMT=QCM
$EXE FF.LMT



        * * *  F U N N E L   F L O W  * * *

INPUT SAMPLE IDENTIFICATION (UP TO 16 CHARACTERS)
ENTER "DONE" AFTER LAST SAMPLE
MANUAL
INPUT THE FUNNEL FLOW TIME FOR MANUAL
20 7
INPUT SAMPLE IDENTIFICATION (UP TO 16 CHARACTERS)
ENTER "DONE" AFTER LAST SAMPLE
DONE
STOP FF


$JOB
$END QCLAB
$
```

43324

21134039

FIGURE 17

```
JOB
$
GCLAB PMT
$ASS 1=TY 3=ATG RAD=GCD LMT=GCM
$EXE PMT.LMT



        * * *  P O W D E R   M I X   T I M E  * * *

INPUT SAMPLE IDENTIFICATION (UP TO 16 CHARACTERS)
ENTER "DONE" AFTER LAST SAMPLE
MANUAL
INPUT THE POWDER MIX TIME FOR MANUAL          IN SECONDS
350
INPUT SAMPLE IDENTIFICATION (UP TO 16 CHARACTERS)
ENTER "DONE" AFTER LAST SAMPLE
DONE
STOP PMT


$JOB
$END GCLAB
$
```