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SERI-PR-8279-1-T1

LOW-COST SILICON PROCESS DEVELOPMENT

Phase IV: Process Improvement

FIRST QUARTERLY
TECHNICAL PROGRESS REPORT

E. Moore
C. Newman

JANUARY 1981

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ABSTRACT

The J. C. Schumacher Company is investigating a number of promising techniques for improving the overall yield and economics of the tribromosilane based process to produce solar cell grade silicon. The current work is aimed at the identification of an optimum process and the characterization of that process through mini-plant operation and analysis. The three project tasks include process improvement studies, kinetic studies, and process economic studies.

During the last quarter process improvement studies in the mini-plant focused on the correlation of current mini-plant yield results with prior laboratory scale work. The silicon product reactor was operated in the thermal disproportionation mode and successfully processed 35 kilograms of tribromosilane. The yield during the test ranged from 75 to 85 percent of theoretical. The experimental apparatus for use in the determination of the decomposition rate of tribromosilane was assembled, tested and placed into service.

PROJECT DESCRIPTION

The goal of the Low Cost Silicon Process Development Project's Process Improvement Phase is three-fold. First, the mini-plant process systems will be utilized to further study various laboratory developments which show good promise of improving overall process economics. The most notable of these developments are the various catalytic agents which have been found to promote the hydrogenation of silicon tetrabromide in the synthesis reactor unit. Secondly, an experimental chemistry task is focused on the determination of the tribromosilane decomposition rate. The results of this rate determination task will be integrated into the mini-plant through design improvements in the product reactor. Lastly, through economic analysis of the various process schemes the most viable production process will be chosen for full process characterization by way of extended mini-plant operations.

SECTION 1

INTRODUCTION

Low cost silicon process development work began in 1977 in the J. C. Schumacher Research Laboratory. The experimental process improvement work described in this report is sponsored by the Solar Energy Research Institute under DOE/SERI Contract XS-9-8279-1. The contract period is June 1979 through February 1981.

1.1 Process Description

The J. C. Schumacher Company's low cost silicon process is based on the silicon-bromine-hydrogen chemical system whose favorable characteristics make possible the generation of a high purity, granular polycrystalline silicon product from metallurgical grade silicon in a closed-loop process. The process chemistry was described in detail in the progress report ⁽¹⁾ issued in October 1979.

1.2 Project Objective

The project goal of this process improvement phase is the characterization and analysis of various process alternatives which show good possibilities for overall yield increases or cost reductions.

Earlier phases of this work focused on the basic process chemistry and the integration of the various processing blocks into a closed-loop system. During this phase, the decomposition rate of tribromosilane will be determined experimentally and certain catalytic reactor configurations which have shown improved yields of tribromosilane in experimental work will be studied in the mini-plant. Production reactor design improvements as indicated by rate determination studies will be incorporated into the mini-plant reactors for verification. Overall, the most viable process, both from technical and economic considerations will be identified for further study in the mini-plant.

1.3 Technical Approach

As described above, the three main areas of technical effort include experimental rate determination studies, the study of processing alternatives and catalysts in the mini-plant, and the selection of the most technically sound and economically viable process for further characterization in the mini-plant.

The results of the rate determination work will be used specifically to develop theoretically based silicon production reactor designs, which, depending upon the true nature of the decomposition reaction, may include physical changes to the reactor and/or modifications to the operating scheme.

The various catalytic agents which have been shown to promote the production of tribromosilane by the hydrogenation of silicon tetrabromide will be studied in the mini-plant in a series of material balance tests. These tests are designed to verify the suitability of a given catalyst system both from the standpoint of the yield of suitable products and by-products as well as from the potential introduction of difficult to remove contaminants in the reactor product. Economic analysis of all technically suitable processes will enable selection of the "best" silicon production process. This process will be subjected to extensive heat and material balance testing in the mini-plant for the purpose of fully characterizing all significant parameters.

SECTION 2

EXPERIMENTAL CHEMISTRY & KINETIC STUDIES

The mini-plant facility synthesis reactor has produced tribromosilane at yields similar to previous laboratory scale work at low temperatures but when high reactor temperatures were used the yields fell below the expected values. The laboratory studies were performed in reactors of 3.0 and 6.0 cm while the pilot studies were conducted in a five inch (12.5 cm) reactor. Experimental scale-up complications in the pilot facility including heat transfer and other transport phenomena might be the cause of this lower yield. Since the hydrogenation of tetrabromosilane is very endothermic, special operational considerations are necessary to increase tribromosilane yields for the larger reactor, a laboratory scale experimental study has been initiated to study the effects of reactor sizing on the yield of tribromosilane.

2.1 Synthesis of Tribromosilane by the Hydrogenation of Tetrabromosilane

A large scale laboratory reactor (~9.0cm) will be employed to study possible reactor scale-up problems and to explore likely solutions to these problems. This laboratory scale study will investigate reaction variables that control the rate of tribromosilane production. The variables to be studied are the effects of hydrogen to tetrabromosilane feed ratio, effects of reactor temperature, effects of reactant residence times and the effects of M.G. silicon particle size. Material balance experiments will also be performed. The additional information obtained from this study will be valuable in present and future tribromosilane reactor scale-up.

2.2. Tribromosilane Thermal Disproportionation Kinetic Study

The silicon product reactor produces high purity silicon by the thermal disproportionation of tribromosilane.

In prior laboratory scale fluidized bed studies of this silicon production reaction 85% of the theoretical was attained. Two pilot plant fluidized bed test runs have recently yielded 75% and 85% of the theoretical yield. Among the potential reasons these reactor tests have not produced silicon at higher efficiency are that less than optimum conditions were used and that some very efficient back reaction may be occurring. The existence of a back reaction could suggest that the overall reaction is at a quasi equilibrium state and the observed tribromosilane in the effluent is the equilibrium product of a hydrogenation of tetrabromosilane reaction. To determine the true nature of the basic yield limiting factors a well controlled kinetic study is essential.

In order to study the kinetics of the tribromosilane thermal disproportionation, the use of a static bulb reactor is planned because of the many process control advantages it has over the fluidized bed reactor. The static reactor has better control over residence time, reaction temperature and the reactor surface area is more precisely defined than a pilot scale fluidized bed reactor. The kinetic results obtained from the static bulb reactor will benefit from the enhanced process control and should provide greater insight into optimizing the product silicon process.

To demonstrate the suitability and the capabilities of the static bulb reactor system, a series of calibration tests will be performed. The accuracy of the system can be determined by observing and reproducing a well characterized molecular decomposition (Table 1). The molecular decomposition chosen to standardize the reactor system is the t-butyl chloride decomposition to 2-methyl-propene and hydrogen chloride.

Comparison of the results of the t-butyl chloride decomposition study with those of previous investigations will illustrate the accuracy of the reactor system and experimental techniques.

Identical experimental procedures will be used in the tribromosilane thermal disproportionation reaction study as done in the t-butyl chloride decomposition study.

TABLE 1

Rate constants for the unimolecular decomposition of t-butyl chloride to 2-methyl-propene and hydrogen chloride.

	RATE CONSTANT (Sec ⁻¹)		Temperature Range (K°)	Reference
10	12.4 ± 0.2	e ^{-41,400 ± 600/RT}	563-614	2
10	13.9 ± 0.7	e ^{-45,000 ± 1900/RT}	547-624	3
10	13.77 ± 0.2	e ^{-45,000 ± 600/RT}	563-603	4
10	13.9	e ^{-46,200/RT}	750-950	5
10	13.74	e ^{-44,690/RT}	715-845	6

2.3 Experimental Procedures

All separations and chemical transfers will be performed in a vacuum system using the techniques outlined by Jolly.⁽⁷⁾ A diagram of the vacuum system is shown in Figure 1.

The vacuum system routinely operates at internal pressures of 10^{-4} mm Hg. These pressures are achieved by using a Duo-Seal, Model 1376B forepump and a single stage mercury diffusion pump, and are measured by a McLeod gauge attached to the main manifold of the vacuum system. Four overhead storage bulbs and four liquid storage bulbs are also attached to the main manifold.

The kinetic reactions will be carried out in a static bulb reactor similar to those of Paquin⁽⁸⁾ and Vanderwielen⁽⁹⁾. The quartz reactors are cylindrical with volumes of 954 ± 25 cc. The reactors are heated with electric resistance heaters and are insulated. Reactor temperatures are measured to the nearest tenth degree centigrade (0.1°C) by a thermocouple connected to a millivolt potentiometer (Leeds and Northrup, Model 8686-3).

Reactant and products will be measured quantitatively by gas chromatography (Carle Inst., AGC-1111 Equipped with a thermal conducting detector). Quantitative measurements of the t-butyl chloride decomposition system and the tribromosilane thermal disproportionation system will be made using a 6' x 1/8", 8% OV101 on Chromosorb W HP 80/100 mesh stainless steel column operating at 23°C and 40°C respectively. With a helium flow rate of about 21 cc/min., retention times for t-butyl chloride, 2-methyl-propene and hydrogen chloride are 1.75, 0.72, and 0.53 minutes respectively. The retention times of tribromosilane and tetrabromosilane, using the same helium flow rate are 5.8 and 23 minutes.

The use of thermal conductivity detectors for quantitative analysis requires relative sensitivity calibrations for each component analyzed. Thus standardized known mixtures of product and reactant gases must be periodically chromatographed to insure stable and reproducible results. Two standardized known mixtures of t-butyl chloride and 2-methyl-propene were prepared and will be analyzed early in the next quarter.

FIGURE 1
EXPERIMENTAL RATE STUDY VACUUM SYSTEM

SECTION 3

PROCESS DEVELOPMENT TESTS

This report covers the calendar quarter ending on 31 December 1980 however, only during the month of December was work performed under this contract. During the period immediately preceding the initiation of the current contract, the experimental mini-plant facility was subjected to extensive start-up tests and certain indicated modifications were implemented. At the beginning of the contract period the mini-plant was fully operational and no open process design modifications were outstanding.

3.1 Tribromosilane Synthesis Unit

The synthesis reactor unit was in operation 55% of the time during the quarter. In mid-month the process was changed from hydrogenation to bromination to make-up inventory. Lost operation time resulted from a closure flange failure which occurred during a reactor loading cycle just prior to the bromination process start-up.

The main function of the synthesis unit, of course, is the regeneration or synthesis of tribromosilane from the silicon tetrabromide recycled from the product reactor unit. Prior experimental work has shown regeneration efficiencies on the order of 30 to 40 percent throughout various combinations of operating parameters. The initial operational testing has focused on experimentally correlating the laboratory results to the scaled-up mini-plant reactor system. Results during the quarter show tribromosilane yields in the range predicted from early small scale studies. The operation results are given in Table 2 and are shown graphically for comparison with the results of the small scale laboratory study in Figure 2.

3.2 Product Reactor Unit

The silicon product reactor was successfully operated during a three day period in December. The results show a 75 to 85 percent yield based on the stoichiometry of the thermal disproportionation reaction. During the test period approximately 35 kilograms of tribromosilane were processed. The operation tests were terminated when the inventory was depleted.

FIGURE 2

MINI-PLANT SYNTHESIS REACTOR - YIELD VERSUS REACTOR TEMPERATURE - NO CATALYST

TABLE 2

OPERATION DATA & RESULTS - SYNTHESIS REACTOR

HYDROGENATION MODE

5" DIA REACTOR

Test Date	H ₂ ℓ/m	SiBr ₄ ml/m ⁴	mole ratio	Reactor Temperature °C	Residence Time sec	SiHBr ₃ % Yield
12/02	30	70	2.2	650	3.8	6.4
12/02	22	35	3.3	650	5.9	9.4
12/02	23	35	3.4	650	5.7	9.0
12/04	16	31	4	650	8.4	11.3
12/08	25	55	2.4	700	4.6	9.4
12/09	16	52	1.6	700	6.3	14.9
12/09	21	52	2.1	700	5.3	14.8
12/09	20	50	2.1	700	5.6	14.2
12/10	20	53	2	700	5.4	13.2
12/10	20	53	2	700	5.4	16.3

3.3 Engineering Support Activities

The first quarter support activities focused on the development and debugging of a computer code designed to enable the plant operator personnel to input operations data on an interactive system to the J. C. Schumacher Co.'s computing facility. The system generates daily operating status reports and files daily results on the Silicon Project data base disk. The code, written in IBM SYSTEM/34 BASIC, is given in the Appendix.

SECTION 4

CONCLUSIONS

Highlights and conclusions of the first quarters work:

4.1 The vacuum system and static-bulb reactor for use in kinetic study was completely assembled.

4.2 The in-line gas chromatograph was placed into operation and standard calibration tasks were initiated.

4.3 An experimental synthesis reactor was designed and construction was begun. Preparation of tetrabromosilane for these tests was also begun.

4.4 Initial operational testing of the synthesis reactor in the low range of process temperatures showed good correlation with prior laboratory results.

4.5 The silicon reactor sustained operation for a three day period and operated at 75 to 85 percent of stoichiometric yield.

4.6 The mini-plant operation analysis and report computer code was placed into general use.

SECTION 5

PLANNED ACTIVITY

5.1 Experimental Chemistry Tasks.

- Initiate material balance tests using large scale laboratory synthesis reactor.
- Complete kinetic calibration studies.
- Start kinetic studies of tribromosilane decomposition.

5.2 Process Development Tasks .

- Operate synthesis unit in high temperature range to gain maximum conversion.
- Install catalytic reactors and start-up operation.
- Continue operation of silicon product reactor.
- Characterization and chemical analysis of silicon product.
- Analysis of separation efficiency of the purification unit.

SECTION 6

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4. S. C. Wong, Ph.D. Thesis, University of London (1958).
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7. W. L. Jolly, The Synthesis and Characterization of Inorganic Compounds, Prentice-Hall, New Jersey (1970).
8. D. P. Paquin, Ph.D. Thesis, University of California, San Diego (1976).
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APPENDIX

PROGRAM NAME: REPORT
SYSTEM/34 BASIC -- RELEASE 06
LISTP

PAGE 0001
TIME 11.34.52
DATE 12/22/80

```
00010 INPUT "ENTER YOUR NAME": NAME$
00020 PRINT #255: NEWPAGE
00030 PRINT #255: DATE$, "OPERATOR ", NAME$
00040 C$="-"
00050 PRINT #255:
00060 INPUT "ENTER SYNTHESIS REACTOR DATA(Y OR N)": ANS$
00070 INPUT "ENTER THE OPERATION DATE (80,DEC,03)": YEAR,MONTH$,DAY
00080 INPUT "WHICH SHIFT DID THE DATA BE TAKEN?(ENTER DAY,SWING,OR NIGHT)": SHIFT$
00090 INPUT "WHAT RUN WAS THIS DATA?(ENTER THE NO.)": RUN
00100 PRINT #255: "THE DATA IS TAKEN ON ",MONTH$,"-",DAY$,"-",YEAR,SHIFT$," SHIFT",RUN," RUN"
00110 IF ANS$="N" THEN 1000
00120 READ S1,S2,S3,S4,S5,S6,S7,S8
00130 OPEN #1: "NAME=ENG5YNI",OUTPUT
00140 PRINT #255: "** SYNTHESIS REACTOR:"
00150 DATA 0,0,0,0,0,0,0,0
00160 OPTION BASE 1
00170 DIM F(50,9)
00180 PRINT #255:
00190 INPUT "THE TEMP OF LOWER ZONE OF SYN RX (10-H-02)?": TEMP1
00200 PRINT #255: "THE TEMP OF LOWER ZONE OF SYN RX(10-H-02) IS",TEMP1,"(C)"
00210 INPUT "THE TEMP OF HIGHER ZONE OF SYN RX (10RXH01)?": TEMP2
00220 PRINT #255: "THE TEMP OF HIGHER ZONE OF SYN RX(10-RXH-01) IS",TEMP2,"(C)"
00230 INPUT "ENTER THE TEMP OF H2 PREHEATER (10H04)": TEMP8
00240 INPUT "ENTER THE TEMP OF FEED PREHEATER (10H03)": TEMP9
00250 PRINT #255: "THE TEMP OF H2 PREHEATER (10H04) IS ",TEMP8,"(C)"
00260 PRINT #255: "THE TEMP OF FEED PREHEATER (10H03) IS",TEMP9,"(C)"
00270 INPUT "FLOW RATE OF H2 (L/MIN)": A
00280 PRINT #255: "THE FLOW RATE OF H2 IS",A,"(L/MIN)"
00290 INPUT "ENTER 1, IF FEED IS BR2; ENTER 2, IF FEED IS SIBR4": M
00300 ON M GOTO 310,370
00310 INPUT "THE FLOW RATE OF BR2(CC/MIN)": B
00320 R=2.26*A/B
00330 VB=.44*B
00340 PRINT #255: "THE FLOW RATE OF BR2 IS",B,"(CC/MIN)"
00350 PRINT #255: "THE RATIO OF H2/BR2 IS",R
00360 GOTO 420
00370 INPUT "THE FLOW RATE OF SIBR4(CC/MIN)": B
00380 R=5.23*A/B
00390 VB=.19*B
00400 PRINT #255: "THE FLOW RATE OF SIBR4 IS ",B,"(CC/MIN)"
00410 PRINT #255: "THE RATIO OF H2/SIBR4 IS",R
00420 INPUT "ENTER THE TOTAL READING NUMBER ": N
00430 INPUT "THE DIAMETER OF THE REACTOR(INCHES)": D
00440 INPUT "THE LENGTH OF LOWER ZONE HEATER (FEET)": L1
00450 INPUT "THE LENGTH OF HIGHER ZONE HEATER (FEET)": L2
00460 V1=3.1416*(D/2)^2*L1*12*2.54^3
00470 V2=3.1416*(D/2)^2*L2*12*2.54^3
00480 Q=(A+VB)*1000
00490 RT1=(V1/(Q*(TEMP1+273)/293))*60
00500 RT2=(V2/(Q*(TEMP2+273)/293))*60
00510 RT=RT1+RT2
00520 AV13=TEMP1
00530 AV14=TEMP2
```


PAGE 0002
TIME 11.34.52
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[illegible]

PROGRAM NAME: REPORT
SYSTEM/34 BASIC -- RELEASE 06
LISTP

PAGE 0003
TIME 11.34.52
DATE 12/22/80

```
01060 PRINT #255:
01070 PRINT #255: "*** DISTILLATION COLUMN:"
01080 PRINT #255:
01090 INPUT "THE TEMP OF REBOIL (20H02)": TEMP3
01100 INPUT "THE TEMP OF PREHEATER (20H01)": TEMP4
01110 PRINT #255: "THE TEMP OF REBOIL (20H02) IS",TEMP3,"(C)"
01120 PRINT #255: "THE TEMP OF PREHEATER (20H01) IS",TEMP4,"(C)"
01130 INPUT "THE PERCENTAGE OF SIBR3(X)": PERC
01140 PRINT #255: "THE PERCENTAGE OF SIBR3 IS",PERC,"(X)"
01150 DIM DF(50,9)
01160 READ SU1,SU2,SU3,SU4,SU5,SU6,SU7,SU8
01170 DATA 0,0,0,0,0,0,0,0
01180 INPUT "ENTER THE TOTAL READING NUMBER": NU
01190 FOR I=1 TO NU
01200 INPUT "THE TEMP OF T5,T6,T7,T22,T26,T27,T28,T30(C)": DF(I,2),DF(I,3),DF(I,4),DF(I,5),DF(I,6),DF(I,7),DF(I,8),DF(I,9)
01210 DF(I,1)=I
01220 SJ1=SU1+DF(I,2)
01230 SU2=SU2+DF(I,3)
01240 SU3=SU3+DF(I,4)
01250 SU4=SU4+DF(I,5)
01260 SU5=SU5+DF(I,6)
01270 SU6=SU6+DF(I,7)
01280 SU7=SU7+DF(I,8)
01290 SU8=SU8+DF(I,9)
01300 NEXT I
01310 AU1=SU1/NU
01320 AU2=SU2/NU
01330 AU3=SU3/NU
01340 AU4=SU4/NU
01350 AU5=SU5/NU
01360 AU6=SU6/NU
01370 AU7=SU7/NU
01380 AU8=SU8/NU
01390 PRINT #255:
01400 PRINT #255:
01410 PRINT #255: " N"," T5"," T6"," T7"," T22"," T26"," T27"," T28"," T30"
01420 FOR I=1 TO NU
01430 PRINT #255: DF(I,1),DF(I,2),DF(I,3),DF(I,4),DF(I,5),DF(I,6),DF(I,7),DF(I,8),DF(I,9)
01440 NEXT I
01450 AU9=TEMP3
01460 AU10=TEMP4
01470 WRITE #2,USING 1480: MONTH1,C$,DAY,C$,YEAR,AU1,AU2,AU3,AU4,AU5,AU6,AU7,AU8,TEMP3,TEMP4,PERC
01480 FORM C 3,C 1,ZD 2,C 1,ZD 2,X 2,N 3,X 2,N 3,X 2,N 3,X 2,N 3,X 2,N 3,X 2,N 3,X 3,N 3,X 4,N 3,X 5,N 4.1
01490 CLOSE #2:
01500 PRINT #255:
01510 PRINT #255: "THE AVERAGE OF THIS SET OF DATA IS:"
01520 PRINT #255:
01530 PRINT #255: TAB(4),AU1,AU2,AU3,AU4,AU5,AU6,AU7,AU8
01540 INPUT "ENTER THE PRODUCT REACTOR DATA (Y OR N)": B$
01550 IF B$="N" THEN 2070
01560 OPEN #3: "NAME=ENGPR01",OUTPUT
01570 PRINT #255: NEWPAGE
01580 PRINT #255:
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PROGRAM NAME: REPORT
SYSTEM/34 BASIC -- RELEASE 06
LISTP

PAGE 0004
TIME 11.34.52
DATE 12/22/80

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01590 PRINT #255:
01600 PRINT #255:
01610 PRINT #255: "***** PRODUCT REACTOR:"
01620 READ SP1,SP2,SP3,SP4
01630 DATA 0,0,0,0
01640 PRINT #255:
01650 INPUT "THE TEMP OF PREHEATER (30H01)": TEMP5
01660 INPUT "ENTER TEMP OF SECOND PREHEATER (30H02)": TEMP6
01670 INPUT "ENTER TEMP OF PRODUCT REACTOR (30RXH01)": TEMP7
01680 INPUT "ENTER THE FLOW RATE OF SIHR3 (CC/MIN)": FS
01690 INPUT "ENTER THE FLOW RATE OF PRODUCT (CC/MIN)": FS1
01700 PRINT #255: "THE TEMP OF PRODUCT REACTOR (30RXH01) IS ",TEMP7,"(C)"
01710 PRINT #255: "THE TEMP OF PREHEATER (30H01) IS ",TEMP5,"(C)"
01720 PRINT #255: "THE TEMP OF PREHEATER (30H02) IS",TEMP6,"(C)"
01730 PRINT #255: "THE FLOW RATE OF SIHR3 IS ",FS,"(CC/MIN)"
01740 PRINT #255: "THE FLOW RATE OF PRODUCT IS",FS1,"(CC/MIN)"
01750 DIM PF(50,5)
01760 INPUT "ENTER THE TOTAL READING NUMBER": NP
01770 FOR I=1 TO NP
01780 INPUT "ENTER THE TEMP OF T15,T16,T17,T19 (C)": PF(I,2),PF(I,3),PF(I,4),PF(I,5)
01790 PF(I,1)=I
01800 SP1=SP1+PF(I,2)
01810 SP2=SP2+PF(I,3)
01820 SP3=SP3+PF(I,4)
01830 SP4=SP4+PF(I,5)
01840 NEXT I
01850 AP1=SP1/NP
01860 AP2=SP2/NP
01870 AP3=SP3/NP
01880 AP4=SP4/NP
01890 INPUT "THE PERCENTAGE OF SIHR3 (%)": PER
01900 PRINT #255: "THE PERCENTAGE OF SIHR3 IS",PER,"(%)"
01910 PRINT #255:
01920 PRINT #255:
01930 PRINT #255: " N"," T15"," T16"," T17"," T19"
01940 FOR I=1 TO NP
01950 PRINT #255: PF(I,1),PF(I,2),PF(I,3),PF(I,4),PF(I,5)
01960 NEXT I
01970 SP5=TEMP5
01980 SP6=TEMP6
01990 SP7=TEMP7
02000 PRINT #255:
02010 WRITE #3,USING 2020: MONTH$,C$,DAY,C$,YEAR,AP1,AP2,AP3,AP4,SP5,SP6,SP7,FS,FS1,PER
02020 FORM C 3,C 1,ZD 2,C 1,ZD 2,X 2,N 3,X 2,N 3,X 2,N 3,X 2,N 4,X 3,N 3,X 3,N 3,X 3,N 4,X 10,N 2,X 11,N 2,X 11,N 4.1
02030 PRINT #255: "THE AVERAGE OF THIS SET OF DATA IS:"
02040 PRINT #255:
02050 PRINT #255: TAB(4),AP1,AP2,AP3,AP4
02060 CLOSE #3:
02070 END
```

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