FGJ A

TO: Distribution

Interoffice Communication

FROM: J.L. Cox, R&D, Ponca City
DATE: April 28, 1987

VISTA

SUBJ: TCLP - ANALYTICAL CAPABILITIES

At the TCLP meeting held in Aberdeen on April 1, questions were raised as to the legality of using Headspace GC for TCLP testing and the precision of VCM analyses. It was decided that I would check on the legal issue and either Ed Sones or I would prepare VCM standards to run at ETC (Memphis), Aberdeen, Oklahoma City and Lake Charles, as well as go to ETC to see if we could offer advice on improving their accuracy.

Joe Ledvina confirmed EPA Method 5020 (Headspace GC) as an acceptable TCLP method. I also spoke with Gayle Hansen at the EPA who said that Method 5020 was acceptable as well as Methods 5030, 8010 and 8240.

On April 2, additional data was available from Ed Sones which showed that while ETC might be having some problems with VCM analysis, there was at least as much error due to loss of VCM from the prepared standards sent to ETC for analysis. With this information, Veldon Messick, Larry Kardos and I decided that it would be better to first determine the precision of Headspace GC for VCM analyses and then, if necessary, look at purge and trap analyses at ETC. Modifications would be looked into, if needed, to improve the precision of the anlysis. Once the precision of the analysis was acceptable, reproducability of standards in the extraction vessels would be checked and, finally, the reproducability of analyzing resin samples.

Results

With only minor modifications on the integration parameters, 30 samples prepared at 50 wppb VCM and 30 samples prepared at 10 wppb VCM in TCLP extraction fluid #1 were analyzed. One 50 wppb sample was thrown out due to incorrect integration. The following results were obtained.

Prepared Conc.	Ave. Observed Concentration	Standard Deviation
50 wppb	48.2 wppb	3.15
10	9.9	0.33

Although the range charts show out-of-control points, the precision is better than was originally expected. The 50 wppb samples had 2 points which were low (31.6 and 30.7 wppb). These could have been due to several reasons other than the instrument. If these 2 points are removed, the average observed concentration is 49.5 wppb with a standard diviation of 1.07 and a range of 46.3 wppb to 51.4. With regard to the 10 wppb, the observed range was 8.8 wppb to 11.1. From this we conclude that our instrumental capabilities in Aberdeen are adequate for TCLP testing.

To check the reproducability of using the extraction vessels, a standard solution using TCLP extraction fluid #1 was prepared and used to fill each of the 8 vessels. In addition, the standard solution was also poured into headspace vials for analysis without going through the additional step of the extraction vessels. This was necessary because VCM loss was unavoidable with the mixing and handling procedures. So, although the intent was to prepare a 40 wppb standard, analyses of the headspace vials showed that the actual concentration was 31 wppb.

After the filled extraction vessels sat 18 hours, 4 samples were taken from each vessel for analysis. Three samples were analyzed that day and one was refrigerated for use later, if needed. The extraction vessels were carried through this procedure twice. VCM analysis on 3 samples from each of the 16 extraction procedures averaged 30.5 or 100% recovery. "$\bar{X}$-R" charting shows this process to be in control. Standard deviation was 1.29.

The reproducability of analyzing resin samples will begin after the rotators arrive in Aberdeen.

Instrumental conditions and SPC charts are attached.

Joyce Cox

Joyce Cox
Chemist

cdh/4-28DIS.JLC
Attachments
Distribution:
Ponca City: SEM HJH JLI ELS
Aberdeen: JF VEM FGJ CWT HGC DSC KWB JWW DWH PJK MLN JGC
OKC: SHC
Houston: JCL LZK
LCVCM: JCC PLF

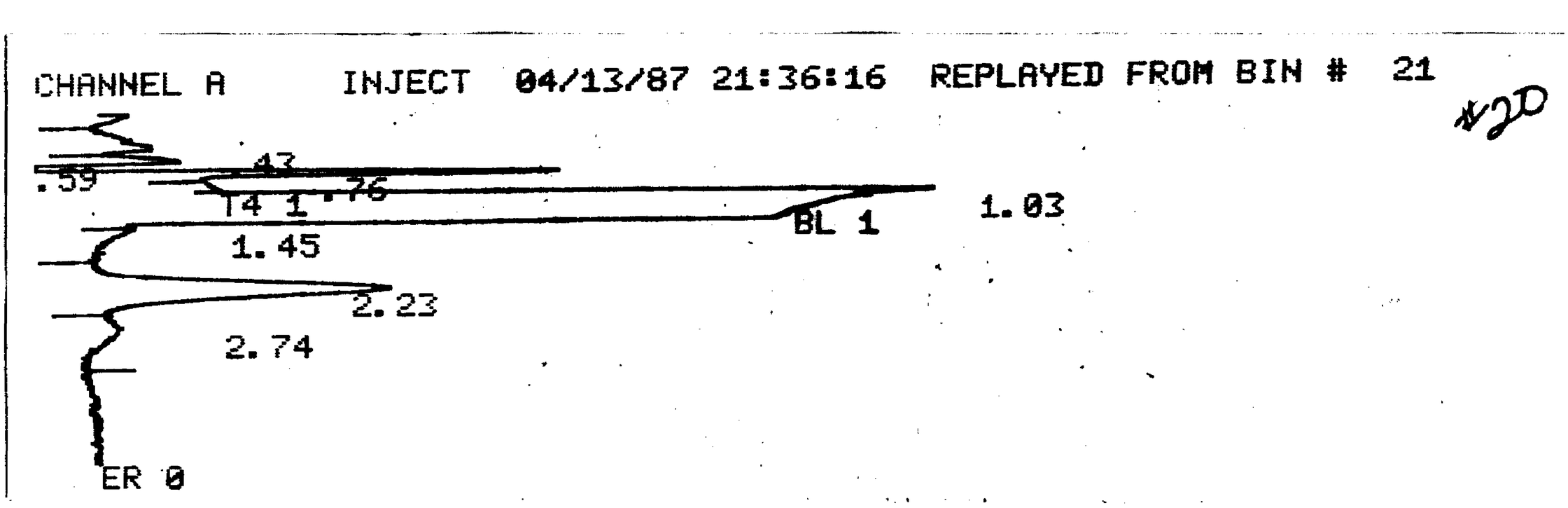
Instrumental Conditions - Perkin-Elmer HS-100

Samples conditioned for 60 min. at 90°C
Prepressurization - 1 min.
Inj. Time - 0.1 min.
Needle Withdrawal - 1 min.
Backflush - Valve on at 1.5 min.
Gas on at 1.3 min.
Gas off at 4.9 min.
Vent - none
Columns - 18" Carbowax 400/Durapak preceding backflush tee
18" Chromosorb 102 after backflush tee
Column Temp. - 119°C
Range - 1
Attenuation - 1
Pressure Gauge Readings
Nigrogen 37/33 psi head/backflush
Air 20
Hydrogen 20

Integration Paramenters - Spectra-Physics 4290

Peak Width - 12
Peak Threshold - 12
BL - on at 1.3 min.

SAMPLE CHROMATOGRAPH



VCM Retention Time - 2.23 minutes
Measured VCM concentration - 18.3 wppb

SPECTRA-PHYSICS

INTEGRATOR FILE

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FILE2

MN=  5.        REM  FE=  2.        CH= "A"  PS=  1.

NM   = "2      "

PW   = 12.          PT   = 12.          CA   = 0.
RN   = 2726.        IX   = 1.           OD   = 2.
PH   = 0.           TB   = 0.           T1   = 1.5
CZ   = 1.           LC   = 0.           LS   = 0.
NV   = 0.           SI   = 1.           SZ   = 1.
RC   = 0.           RA   = 1.           DP   = 0.

TT(1)=     1.       TF(1)="T4"       TV(1)=     1.
TT(2)=     1.3      TF(2)="BL"       TV(2)=     1.
TT(3)=     4.5      TF(3)="ER"       TV(3)=     1.
TT(4)=     4.9      TF(4)="T4"       TV(4)=     0.

RT(1)=     2.23     CN(1)= "RVCMX"     CM(1)= "PPM   "
RF(1)=48000.

AN   = "DTECH"      AN(1)= "      "     AN(2)= "      "

CU   = "      "     CU(1)= "PPM   "

SN(1)= "R     "
SA(1)=     1.       XF(1)=     1.
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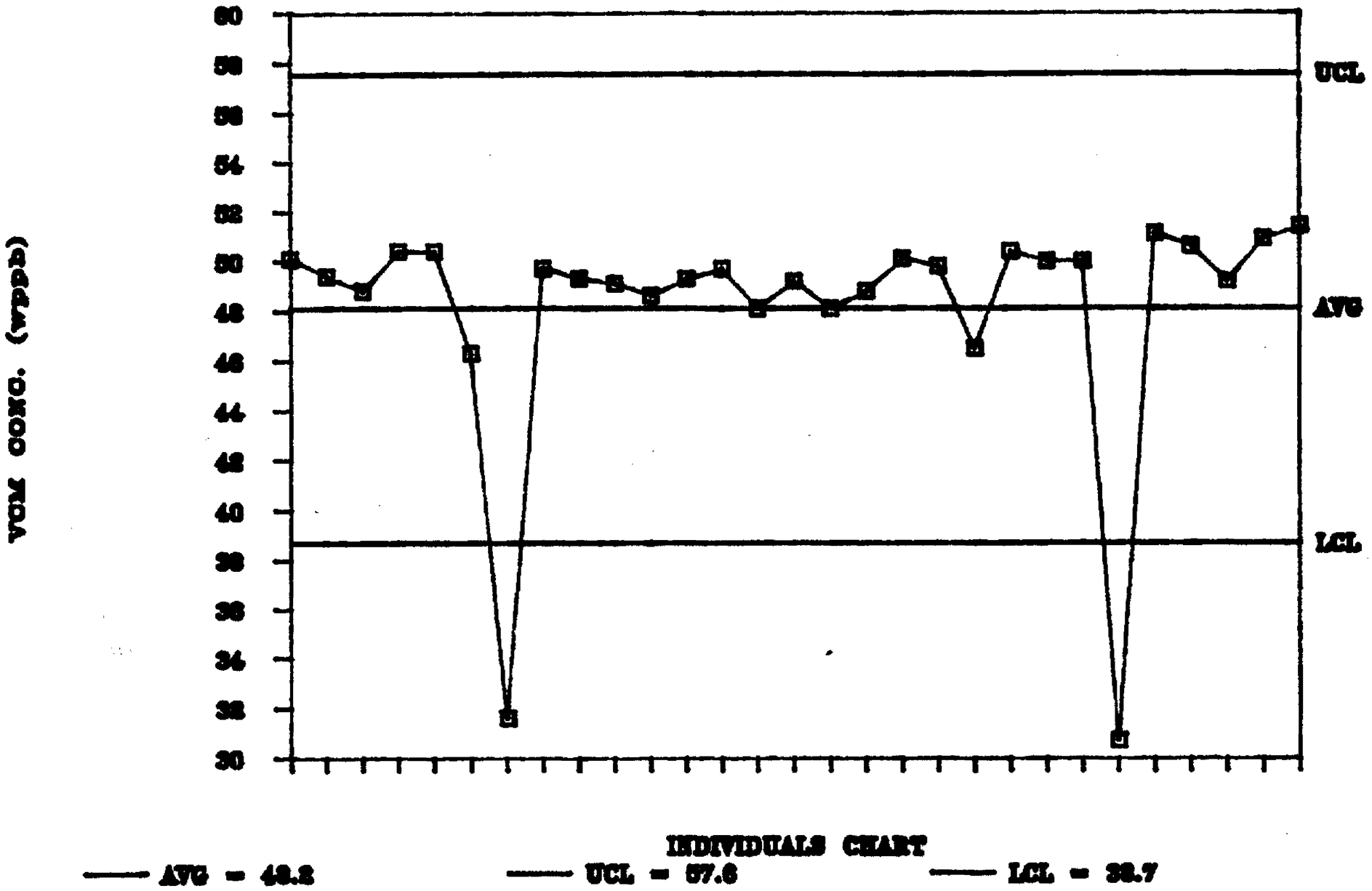
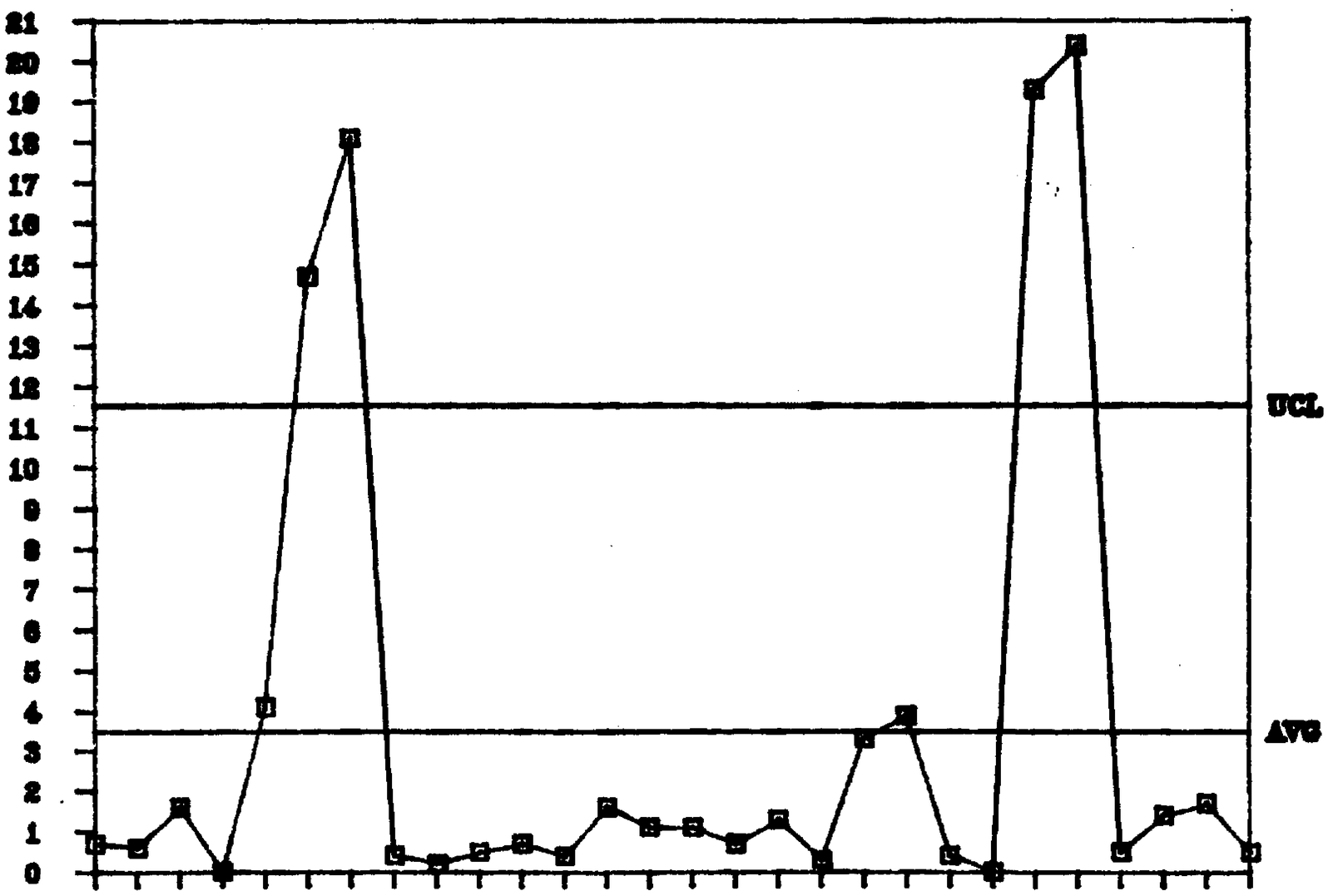
PRECISION TESTING---50 wppb VCM

4/12/87



INDIVIDUALS CHART

—— AVG = 48.2 —— UCL = 57.6 —— LCL = 38.7



R-CHART

—— AVG = 3.8 —— UCL = 11.8

VAB.0001104326

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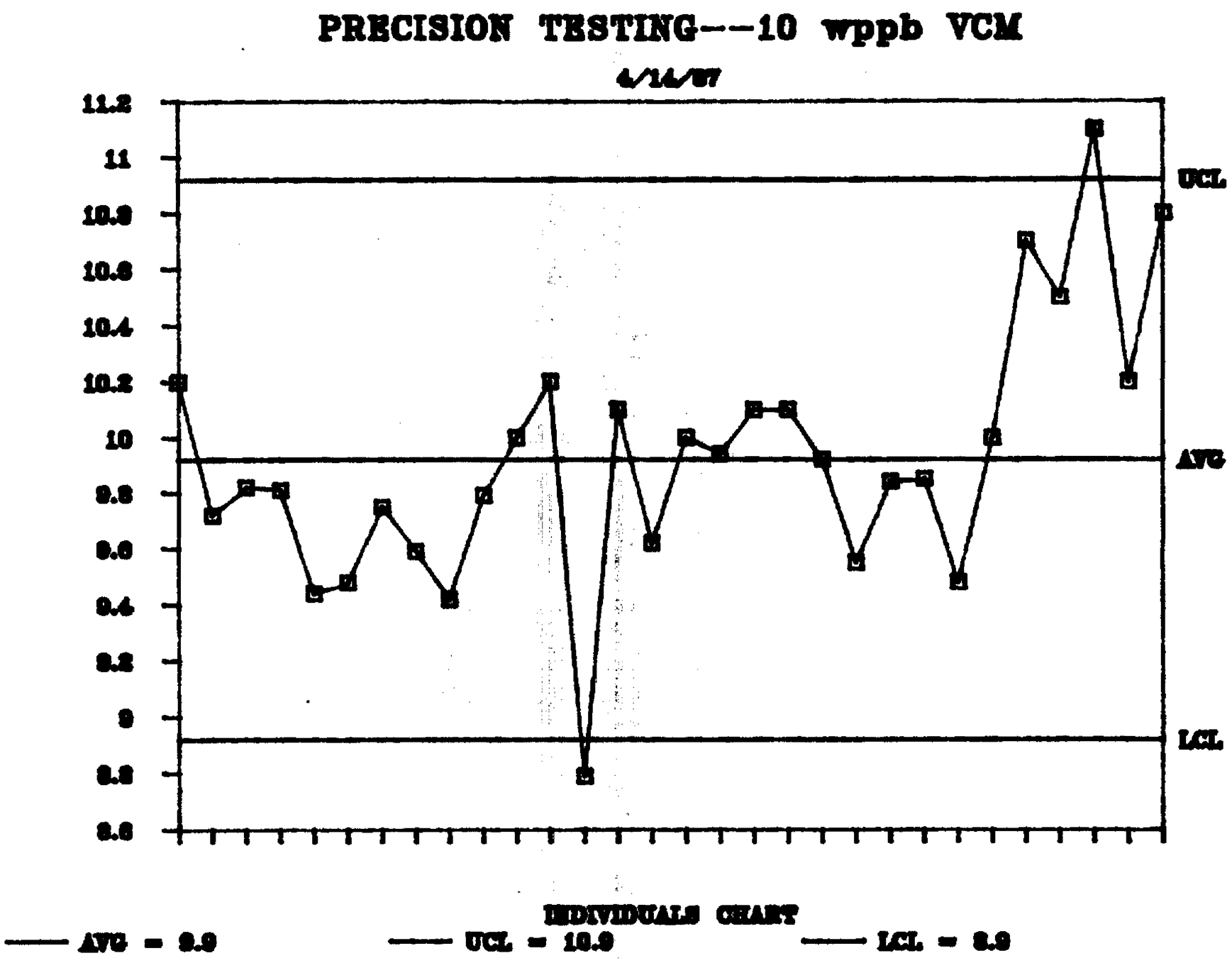
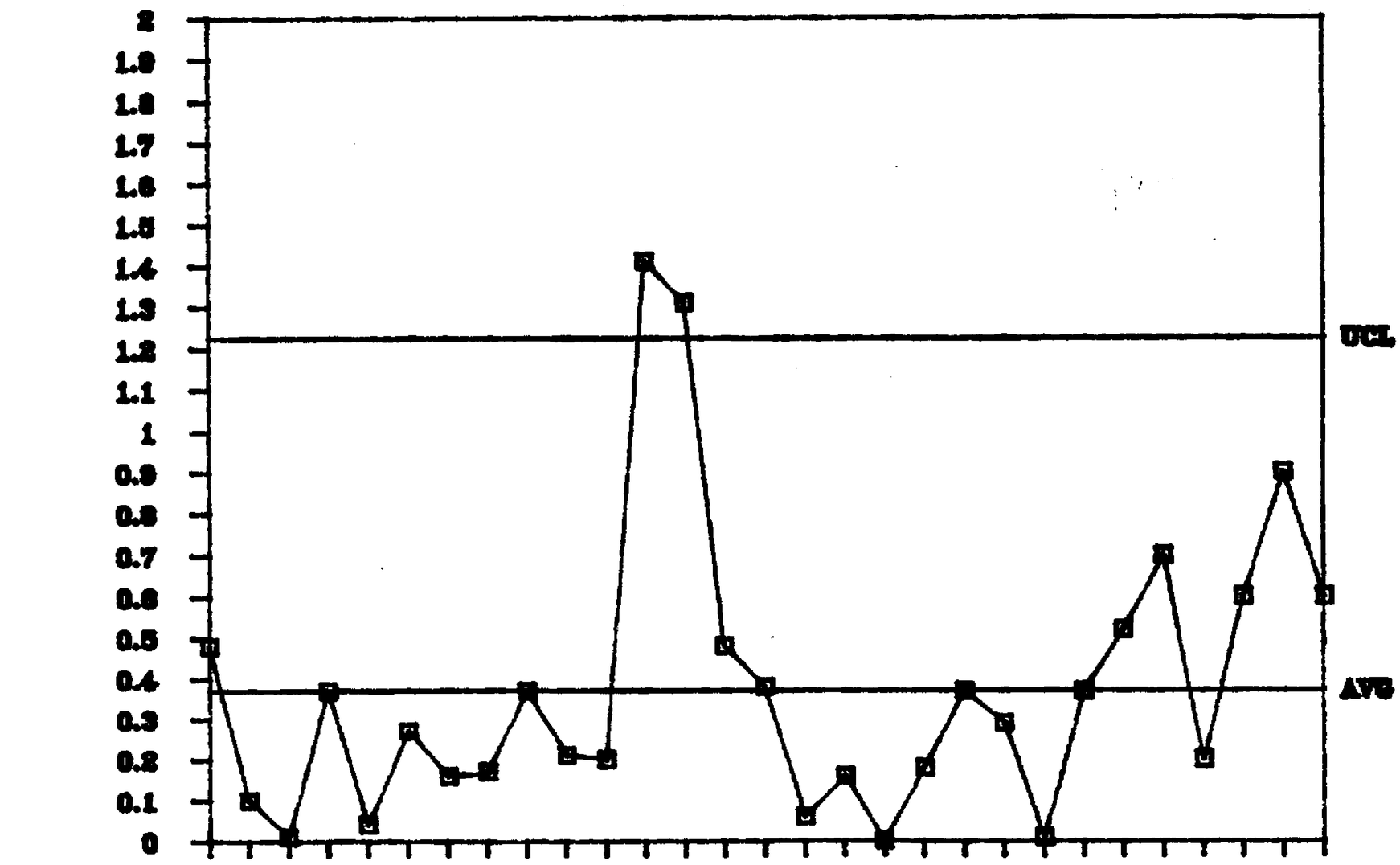

PRECISION TESTING---10 wppb VCM
4/14/87
VCM CONC. (wppb)
11.2
11
10.8
10.6
10.4
10.2
10
9.8
9.6
9.4
9.2
9
8.8
8.6
UCL
AVG
LCL
INDIVIDUALS CHART
AVG = 9.9
UCL = 10.9
LCL = 8.9


2
1.9
1.8
1.7
1.6
1.5
1.4
1.3
1.2
1.1
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
UCL
AVG
R-CHART
AVG = 0.4
UCL = 1.2