Technical Service Report

Conoco Inc.
Research and Development Department
Analytical Research Section
Ponca City, Oklahoma

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Report No. 240-82-814-23C-486

To C. L. Muth

From T. A. Snow

Date March 4, 1982

Subject Quantitative Characterization of VCM Light Ends, Tar Product to PPG and Tars from Still Pump-Out

OBJECT

To characterize samples 4409-107-1 (Tar Product to PPG), 4409-107-2 (Tars from still pump-out) and 4409-107-3 (VCM Light Ends).

CONCLUSIONS

The lightest fraction, sample no. 4409-107-3, contained chlorinated C_1, C_2, C_3 compounds and chlorobutadiene. The other two samples contained chlorinated C_2, C_3, C_4, C_6 and C_8 compounds. A major component list for each of the samples can be seen in Tables I, II and IV. Due to the complexity of the heavier samples (Nos. 4409-107-1 and 4409-107-2) a detailed list of components can be found in Tables III and V.

DISCUSSION

The GC trace is found for the samples in Figures 1, 2, and 9.

Various single ion chromatograms (SIC) were used to show the location of selected classes of chlorinated compounds.

Figures 3 and 9 show the SIC's of 122, 124, 126, which were chosen to show the locations of dichlorobutadienes, dichlorobutenes, trichlorobutenes and trichlorobutanes.

Figures 4 and 10 show the SIC's of 156, 158 and 160, which were chosen to show the location of trichlorobutadienes, trichlorobutenes, tetrachlorobutenes, and tetrachlorobutanes.

Figures 5 and 11 show the locations of mono-, di-, and trichlorobenzenes.

Figures 6 and 12 distinguish the trichlorobenzenes (m/e 180), trichlorohexenes or tetrachlorohexenes (m/e 186), tetrachlorobutadienes or pentachlorobutenes (m/e 190) and tetrachlorobutenes or pentachlorobutanes (m/e 192).

Figure 13 shows the tetrachlorobenzenes (m/e 214), tetrachlorohexane or pentachlorohexane (m/e 220) and pentachlorobutenes or hexachlorobutane (m/e 224).

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Finally the Figures 7 and 14 show chlorinated C_8's (m/e 207), pentachlorobenzene (m/e 248) and hexachlorobenzene (m/e 282).

EXPERIMENTAL

Mass Spectral data was obtained on the Finnigan 4023 GC/MS system. All three samples were split injected (.5 ul) onto an SE30 capillary column. Sample no. 4409-107-3 was injected at 0°C followed by a temperature programming to 100°C at a rate of 4° per minute and 100°C to 260° at a rate of 8° per minute. Sample nos. 4409-107-1 and 4409-107-2 were injected at 20°C followed by a temperature programming to 100°C at 16° per minute and from 100°C to 260°C at 8° per minute. Spectra were obtained at 0.5 second.

REFERENCE

File of MS data: G3159A
G3160A
G3161A

TSR no. 249-80-839-1, "The GCMS Analysis of the Liquid Phase from EDC Tar Solids," Keen, G. W. (1980).

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Chemist
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TABLE I

Components of VCM Light Ends
Sample No. 4409-107-3

Peak No.	Scan No.	Component	% of Total
1	40	Chloroethene	3.4
2	43	Chloroethane with trace of butenol	12
3	50	1,1-Dichloroethene	3.0
4	52	Methylene chloride	1.3
5	57	1,2-Dichloroethene (cis)	4.0
6	59	1,1-Dichloroethane	7.6
7	64	2-Chlorobutadiene	1.4
8	66	1,2-Dichloroethene (trans)	6.1
9	71	Chloroform	24
10	80	1,2-Dichloroethane	19
11	103	Trichloroethene	16
12	103	Trichloroethene	0.35

+ See Figure 1

Table II

Major Components in
Sample No. 4409-107-1
VCM TAR product

Component	% of Total
Dichlorobutenes	11
Trichlorobutenes	12
Dichlorobutadienes	8.7
Tetrachlorobutanes	16
1,2-Dichloroethane	14
C_3-Dichlorobenzenes	1.9
Trichlorohexenes	5.8
Naphthalenes and Indenes	1.7
C_2-Chlorobenzenes	4.3
Trichloroethanes	3.5
C_2-Dichlorobenzenes	8.5
Tetrachloropropanes	1.1
Chlorobenzene	6.3

Total of major compounds is approximately 95% of sample.

Balance of sample can be seen on Table III.

TABLE III
VCM TAR Product
Components of Sample No. 4409-107-1

Peak No.	Scan No.	Component	% of Total
1	48	1,2-Dichloroethane	14
2	61	1,1,2-Trichloroethane	3.5
3	66	3,4-Dichlorobutadiene w/trace dichlorobutene	0.21
4	70	1,4-Dichlorobutadiene w/trace dichlorobutene	8.5
5	76	1,2-Dichlorobutene	6.3
6	83	2,3-Dichlorobutene (2)	11
7	89	Trichlorobutene	5.6
8	99	Trichlorobutadiene	3.0
9	106	Trichlorobutene	3.2
10	110	Chlorinated C_4	0.42
11	118	Tetrachloropropane	1.1
12	124	Tetrachlorobutenes	0.42
13	137	Tetrachlorobutenes	0.42
14	145	Chlorohexadiene	0.29
15	153	Dichlorobenzene	0.44
16	159	Chlorinated C_8	0.17
17	172	Tetrachlorobutane	15
18	177	Tetrachlorobutane	0.80
19	181	Indene with a trace of chlorinated C_4	0.52
20	194	Chloroxylene	2.8
21	204	Trichlorohexene	1.9
22	209	Trichlorohexene	2.8
23	221	Trichlorohexene	1.1
24	226	Chloroethylbenzene	1.3
25	233	Chloromethyltoluene	0.77
26	249	Chlorinated C_8	0.15
27	255	Chlorostyrene	0.13
28	263	Methylindene	0.38
29	273	Chlorinated C_8	0.06
30	284	C_2-Dichlorobenzene	6.7
31	292	Napthalene	0.77
32	301	Dichlorotoluene	0.07
33	307	Chlorinated C_8	0.05
34	322	Chlorobiphenyl	0.88
35	333	C_2-Dichlorobenzene	1.5
36	338	C_3-Dichlorobenzene	1.1
37	348	C_3-Dichlorobenzene	0.80
38	358	Chlorinated compound	0.35
39	368	Chlorinated compound	0.04
40	378	Chlorinated compound	0.06

Table III Continued

Peak No.	Scan No.	Component	% of Total
41	390	Chlorinated compound	0.08
42	396	Dichloroethylbenzene	0.11
43	402	Chlorinated compound	0.10
44	415	Chlorinated compound	0.08
45	430	Chlorinated C_8	0.09
46	449	Bromocyclopentadiene	0.08
47	462	Bromocyclopentadiene	0.15
48	471	Chlorinated compound	0.05
49	483	Ethylmethylstyrene	0.03
50	489	Chlorinated C_6	0.27
51	545	Chlorinated compound	0.03
52	563	Chlorinated compound	0.14
53	574	Chlorinated compound	0.09
54	584	Chlorinated compound	0.18
55	602	Chlorinated compound	0.04
56	622	Chlorinated compound	0.11
57	656	Fluorene	0.10
58	680	Chlorinated	0.05
59	687	Chlorinated compound	0.07

TABLE IV

Major Components In Sample 4409-107-2

TAR still pump out

Component	% of Total
Trichlorobutenes	24
1,1,2-Trichloroethane	18
Dichlorobutadienes	11
Chlorobenzenes	6.3
Dichlorobenzenes	6.6
Dichlorobutenes	1.3
1,1,2,2-Tetrachloroethane	1.8
Tetrachlorobutanes	3.7
Dichlorobutanes	0.8
Pentachloroethane	1.9
Bischloroethylether	1.7
Trichlorobenzenes	5.5
Tetrachlorobutenes	1.8
1,2-Dichloroethane	4.9

Total of Major Compounds is approximately 90% of sample.

Balance of sample is chlorinated compounds. See Table V.

TABLE V

Components of Sample No. 4409-170-2

TAR still pump out

Peak No.	Scan No.	Component	% of Total
1	47	1,2-Dichloroethane	4.9
2	55	1-Bromo-2-chloroethane	0.16
3	65	1,1,2-Trichloroethane	18
4	71	1,4-Dichloro-butadiene	3.8
5	78	2,3-Dichlorobutadiene w/trace of dichlorobutene	5.3
6	83	Chlorobenzene w/dichlorobutadiene	6.3
7	89	Dichlorobutadiene	1.8
8	101	1,1,2,2-Tetrachloroethane	1.8
9	106	Dichlorobutene	1.3
10	116	Chlorinated compound	0.05
11	119	Dichlorobutadiene	0.80
12	132	Trichlorobutadiene	0.72
13	135	Pentachloroethane	1.9
14	138	Bischloroethylether	1.7
15	140	Trichlorobutene	2.4
16	148	Trichlorobutene	3.0
17	155	Trichlorobutene w/dichlorobenzene	0.94
18	169	Dichlorobenzene	6.6
19	194	Trichlorobutene with trace of chloroxylene	18
20	199	Chloroxylene	0.82
21	207	Chlorinated compound	0.59
22	212	Tetrachlorobutene	1.8
23	222	Chlorinated compound	0.45
24	227	Chloroethylbenzene	0.49
25	234	Chloronated compound	0.42
26	239	Chloronated compound	0.11
27	253	Tetrachlorobutane	3.7
28	266	Chloronated C_4	0.12
29	278	Trichlorobenzene	2.7
30	284	Trichlorobenzene w/tetrachlorobutane	2.8
31	292	Napthalene	0.35
32	301	Chlorinated compound	0.02
33	315	Trichlorobenzene	0.04

Peak No.	Scan No.	Component	% of Total
34	322	Chlorinated C_6 and C_4	0.32
35	332	Chlorinated C_8 and C_4	0.70
36	340	Pentachloropropane	0.70
37	347	Chlorinated C_6	0.26
38	359	Chlorinated compound	0.14
39	379	Chlorinated compound	0.02
40	383	Chlorinated compound	0.06
41	392	Chlorinated C_4	0.07
42	402	Chlorinated compound	0.04
43	409	Chlorinated compound	0.05
44	417	Chlorinated compound	0.05
45	431	Dibromochloropropene	0.74
46	448	Chlorinated compound	0.03
47	463	Chlorinated C_6	0.18
48	475	Tetrachlorobenzene	0.03
49	489	Tetrachlorobenzene	0.20
50	491	Tetrachlorobenzene	0.20
51	517	Chlorinated compound	0.03
52	529	Chlorinated C_8	0.01
53	538	Chlorinated compound	0.32
54	546	Chlorinated compound	0.13
55	556	chlorinated compound	0.04
56	562	Chlorinated compound	0.03
57	569	Chlorinated compound	0.04
58	580	Chlorinated compound	0.76
59	603	Pentachlorobenzene	0.37
60	626	Chlorinated compound	0.15
61	642	Chlorinated compound	0.04
62	644	chlorinated compound	0.04
63	649	Chlorinated C_8	0.05
64	653	Chlorinated C_8	0.04
65	659	Chlorinated compounds	0.03
66	659	Chlorinated compounds	0.05
67	669	Chlorinated compound	0.04
68	672	Chlorinated compound	0.03
69	685	Chlorinated compound	0.03
70	688	Chlorinated C_8	0.03
71	701	Chlorinated C_8	0.03
72	704	Chlorinated C_8	0.03

Figure 1. Total Ion Chromatogram for Sample No. 4409-107-2

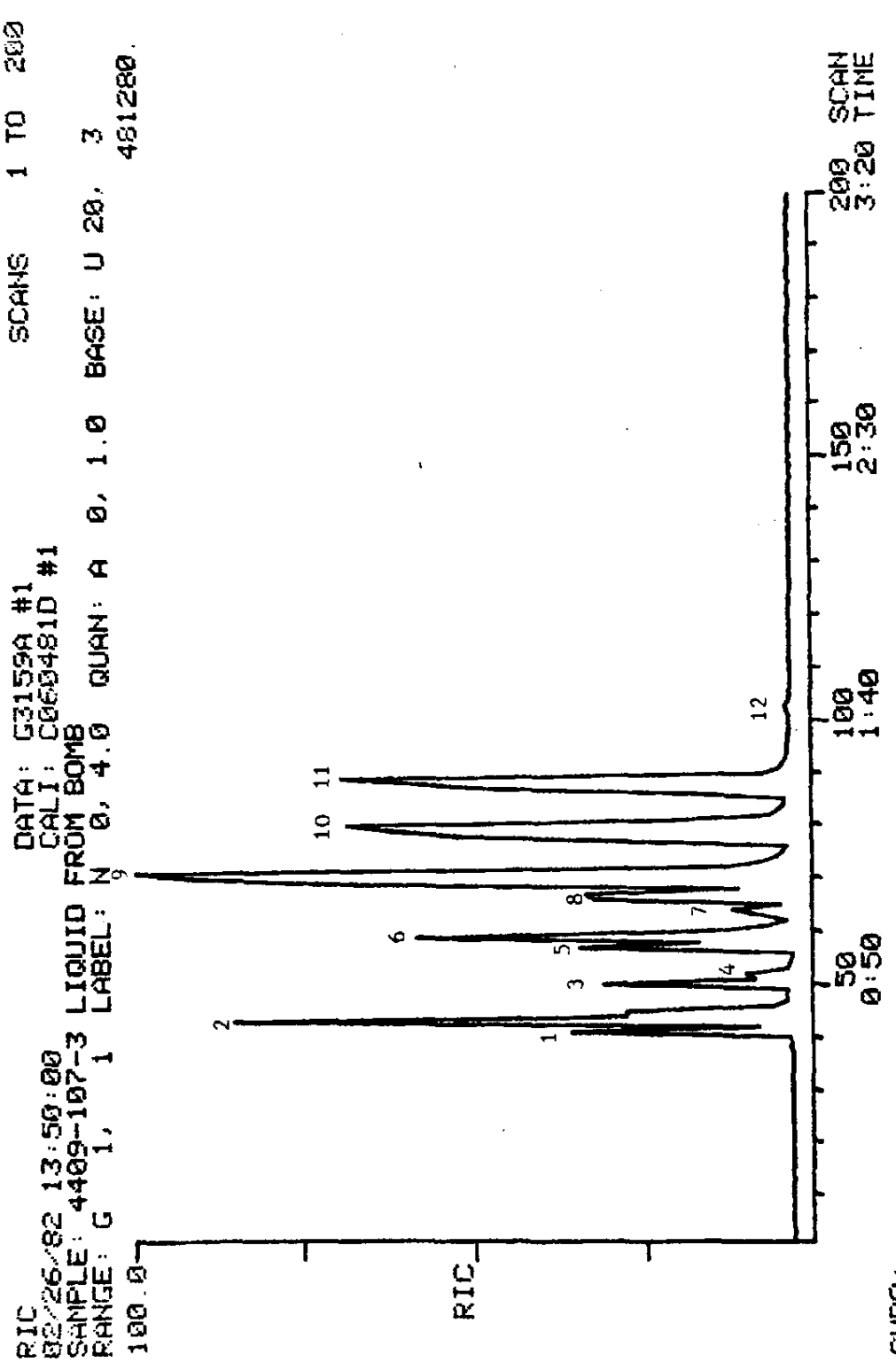

Figure 2. Total Ion Chromatogram of Sample No. 4409-107-1

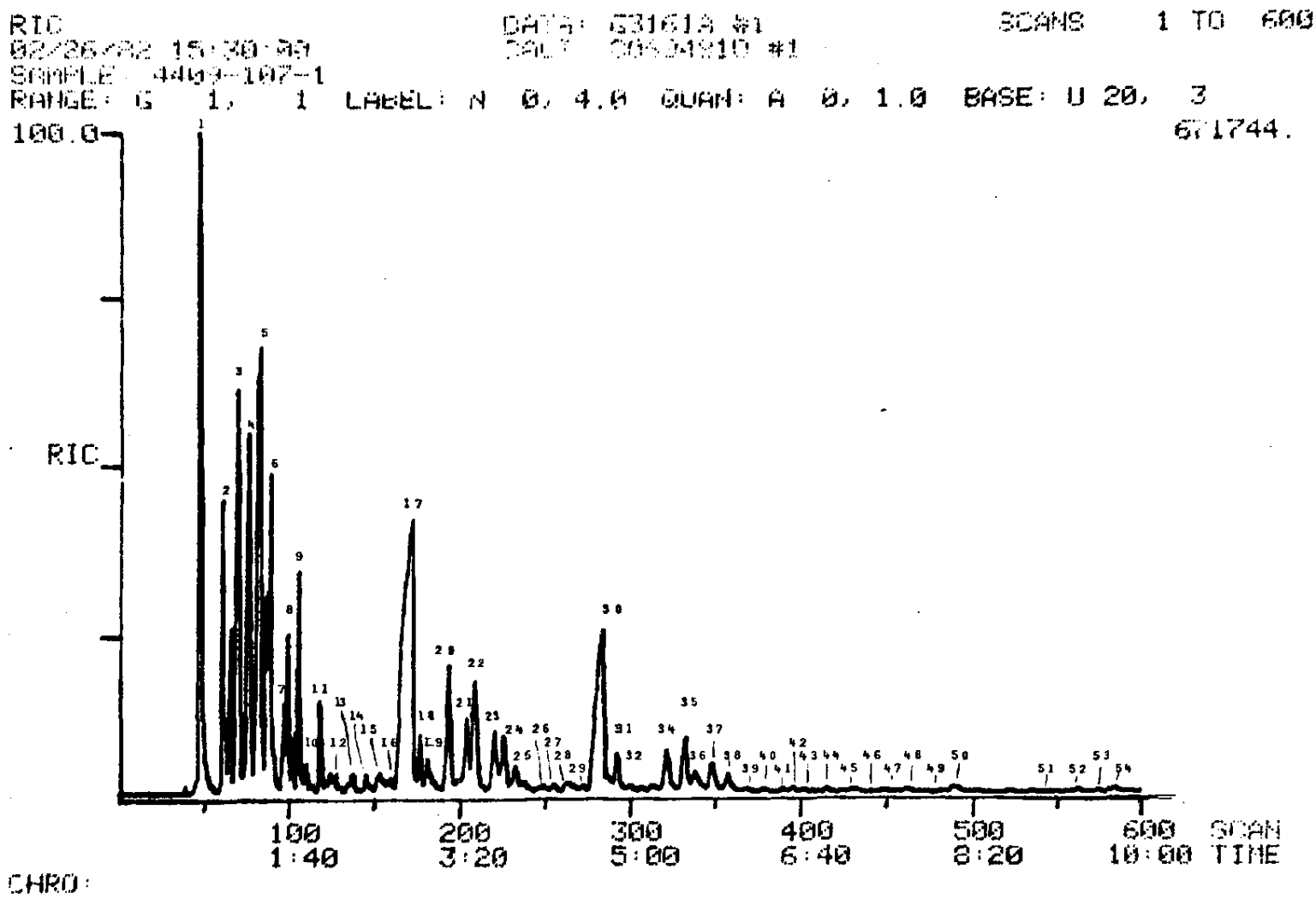

Figure 3. Single Ion Chromatogram sof m/e 122, 124, 126 and Total Ion Chromatogram of Sample 4409-107-1

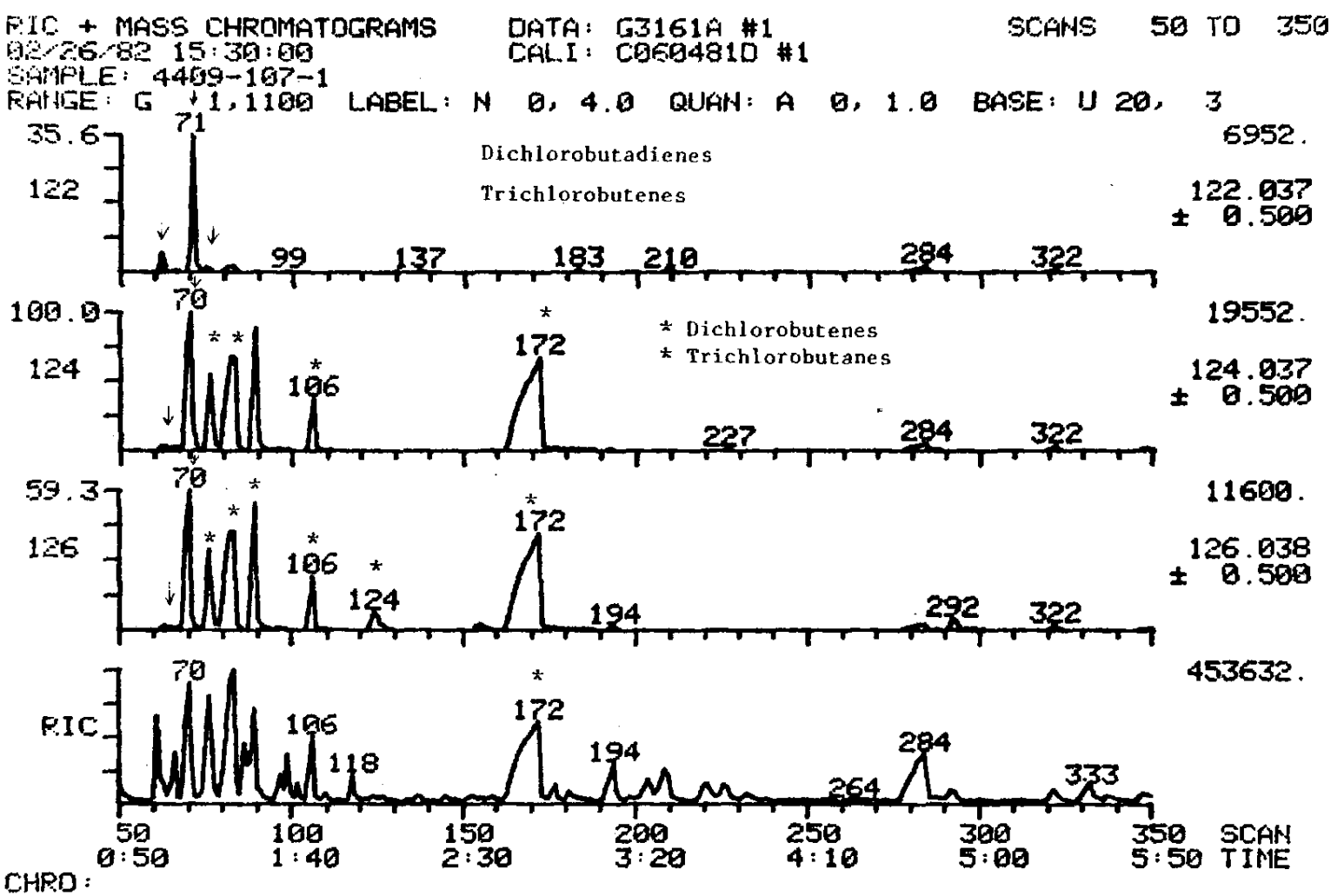

Figure 4. Single Ion Chromatograms of m/e 156, 158, 160 and Total Ion Chromatogram for Sample No. 4409-107-1

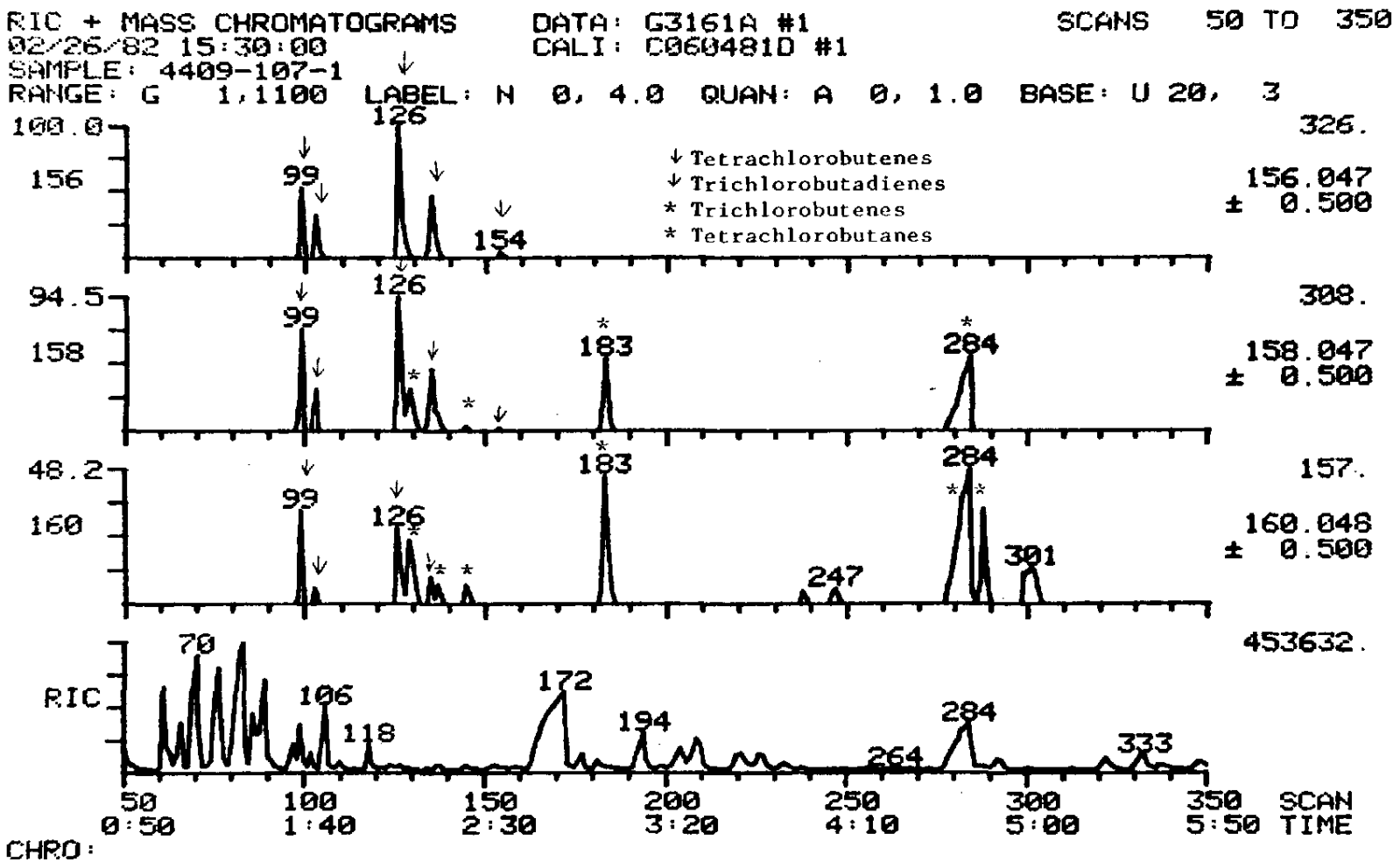

Figure 5. Single Ion Chromatograms of m/e 112, 146, 180 and the Total Ion Chromatogram of Sample No. 4409-102-1

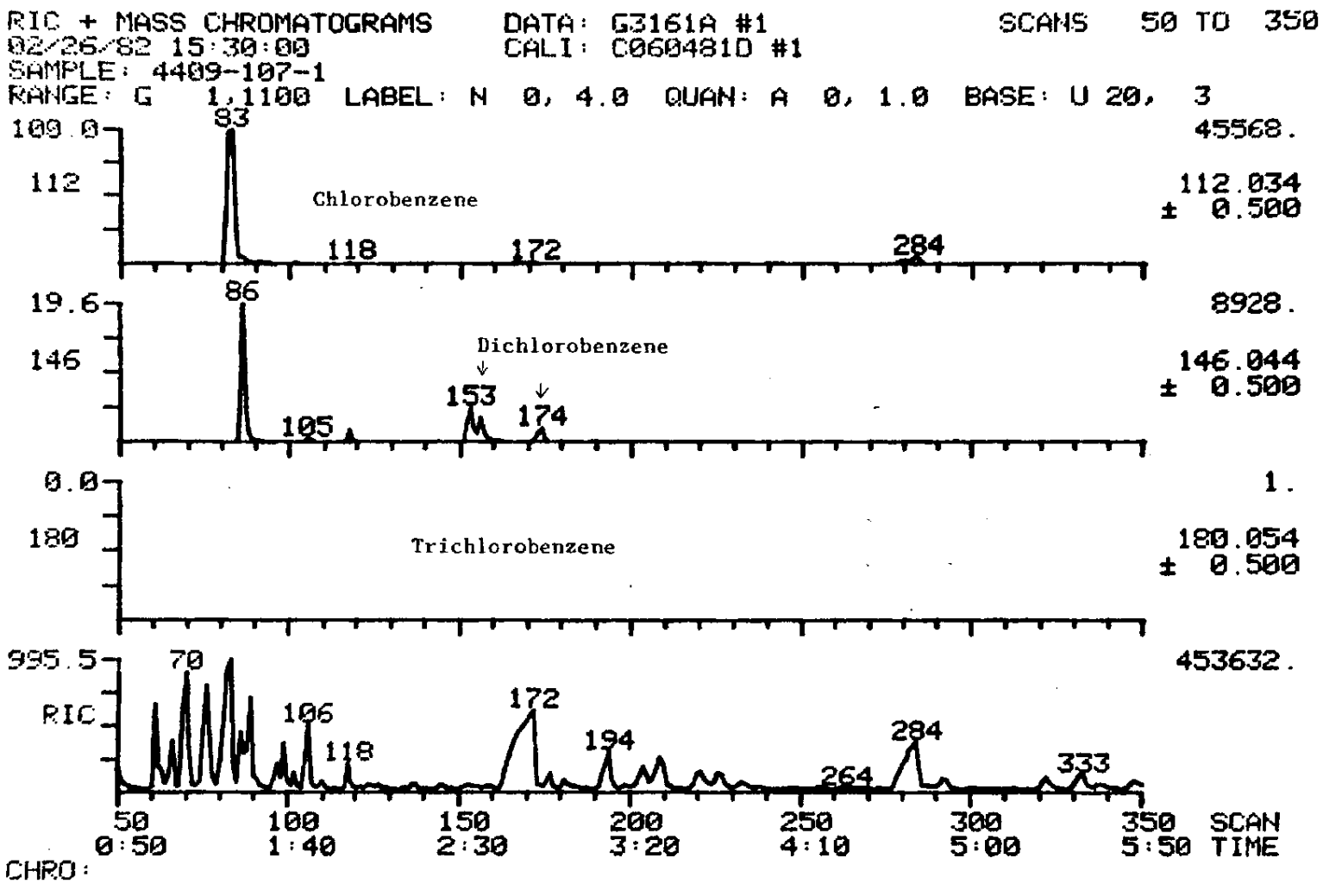

Figure 6. Single Ion Chromatograms of m/e 184, 186, 190, 192 and the Total Ion Chromatogram for Sample No. 4409-107-1

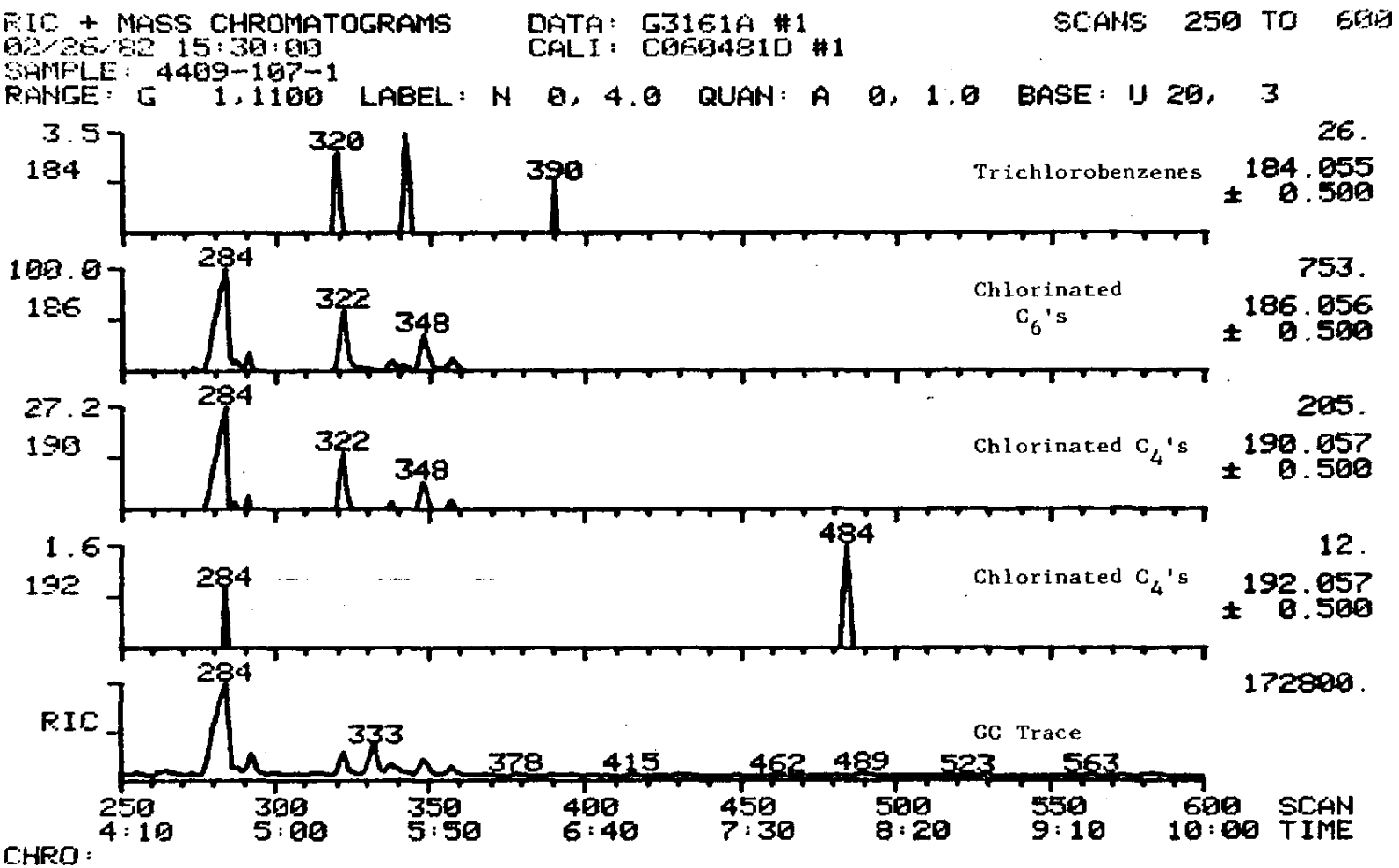

Figure 7. Single Ion Chromatograms of m/e 207, 209, 244, 248 and the Total Ion Chromatogram

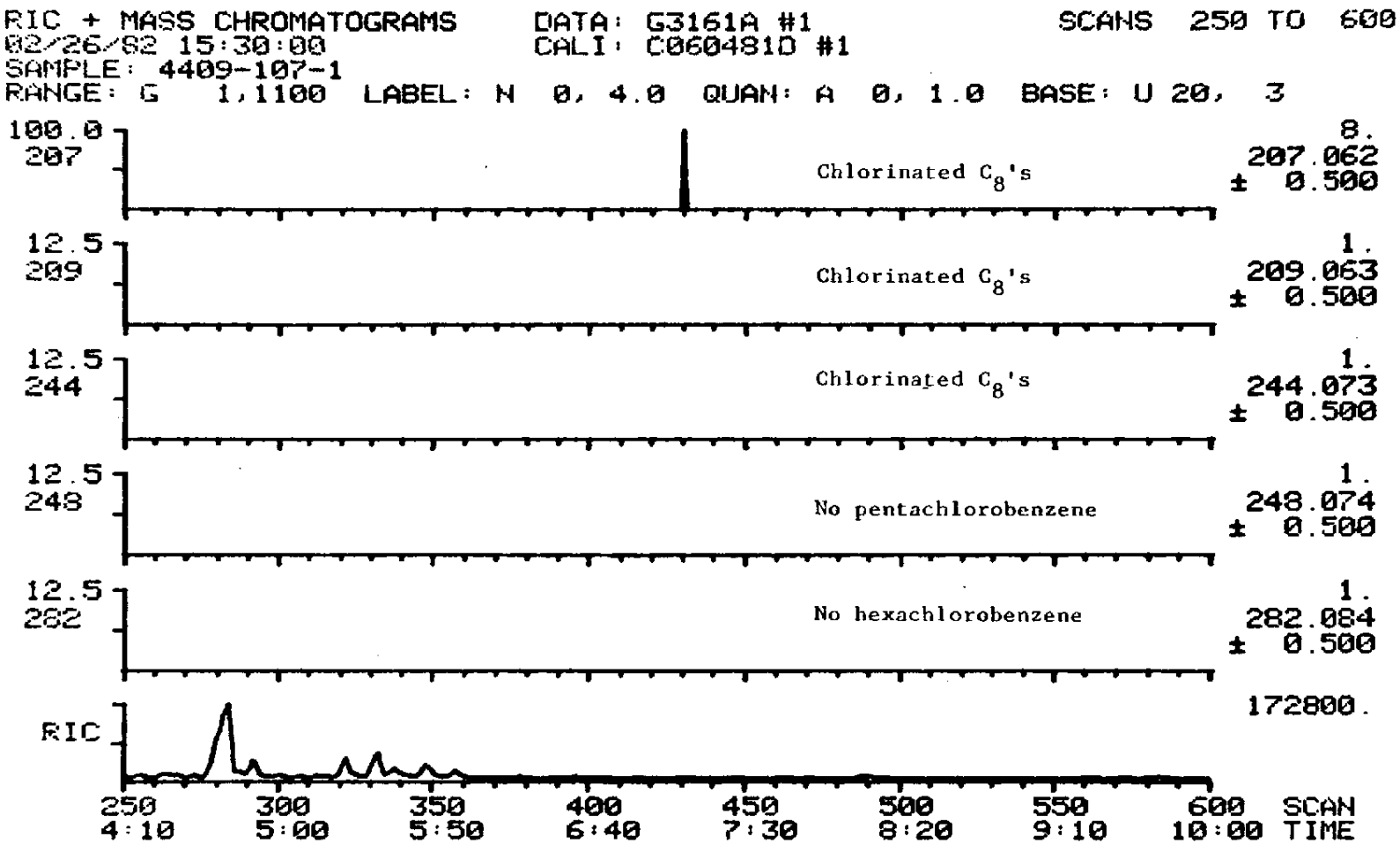

Figure 8. Total Ion Chromatogram of Sample No. 5509-107-2

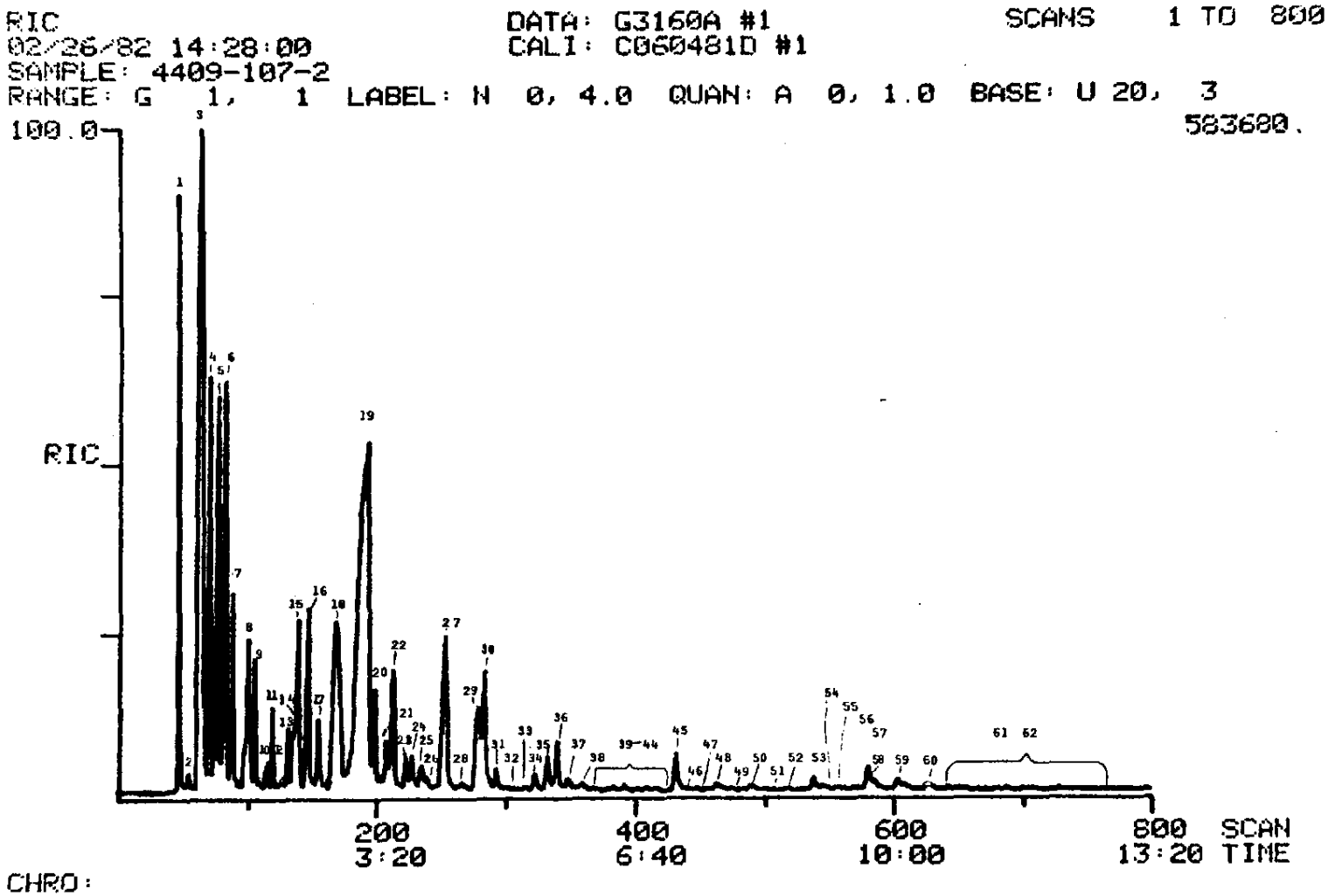

Figure 9. Single Ion Chromatograms of m/e 122, 124, 126 and Total Ion Chromatogram for Sampel No. 4409-107-2

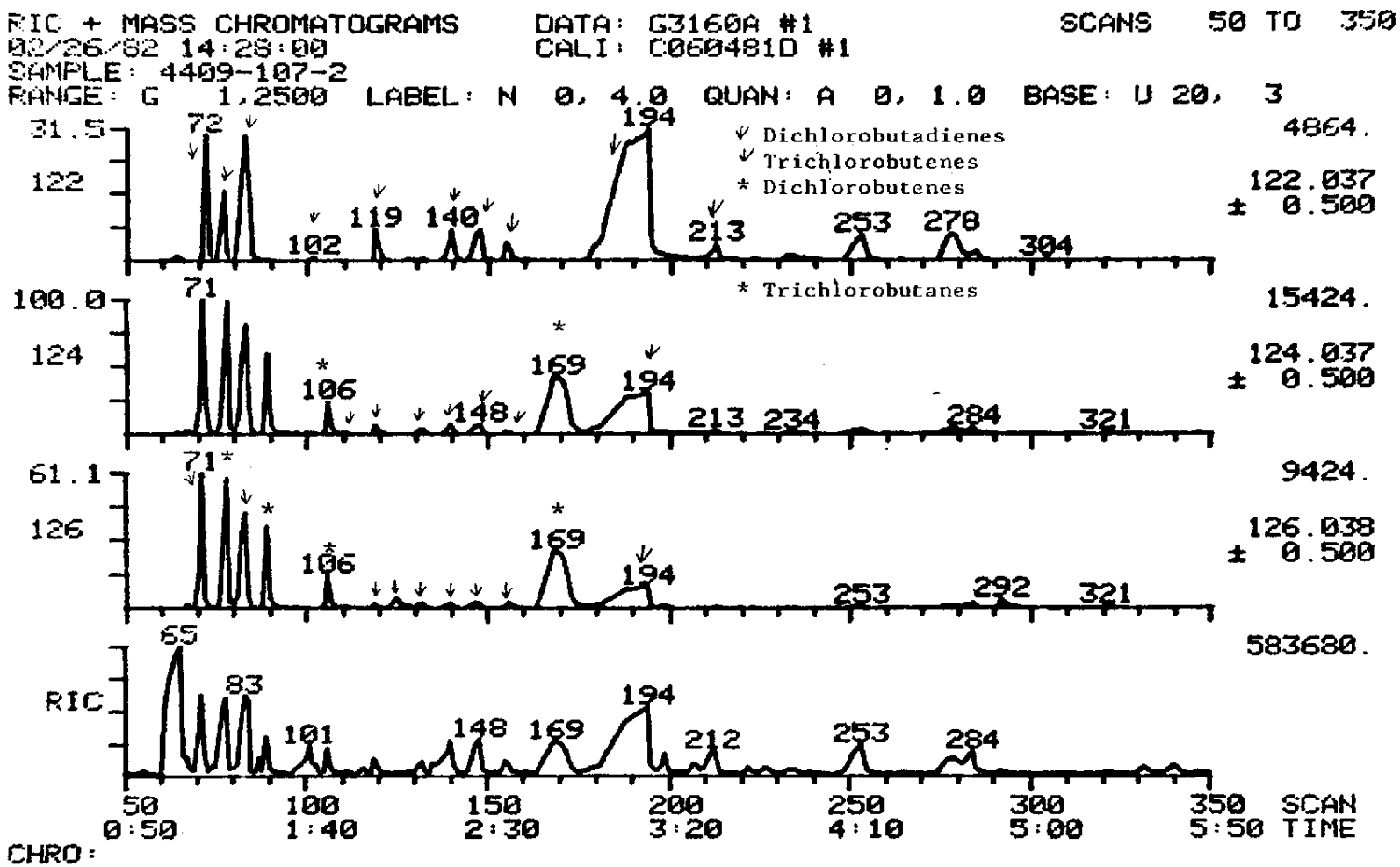

Figure 10. Single Ion Chromatogram of m/e 156, 158, 160 and the Total Ion Chromatogram for Sample No. 4409-107-2

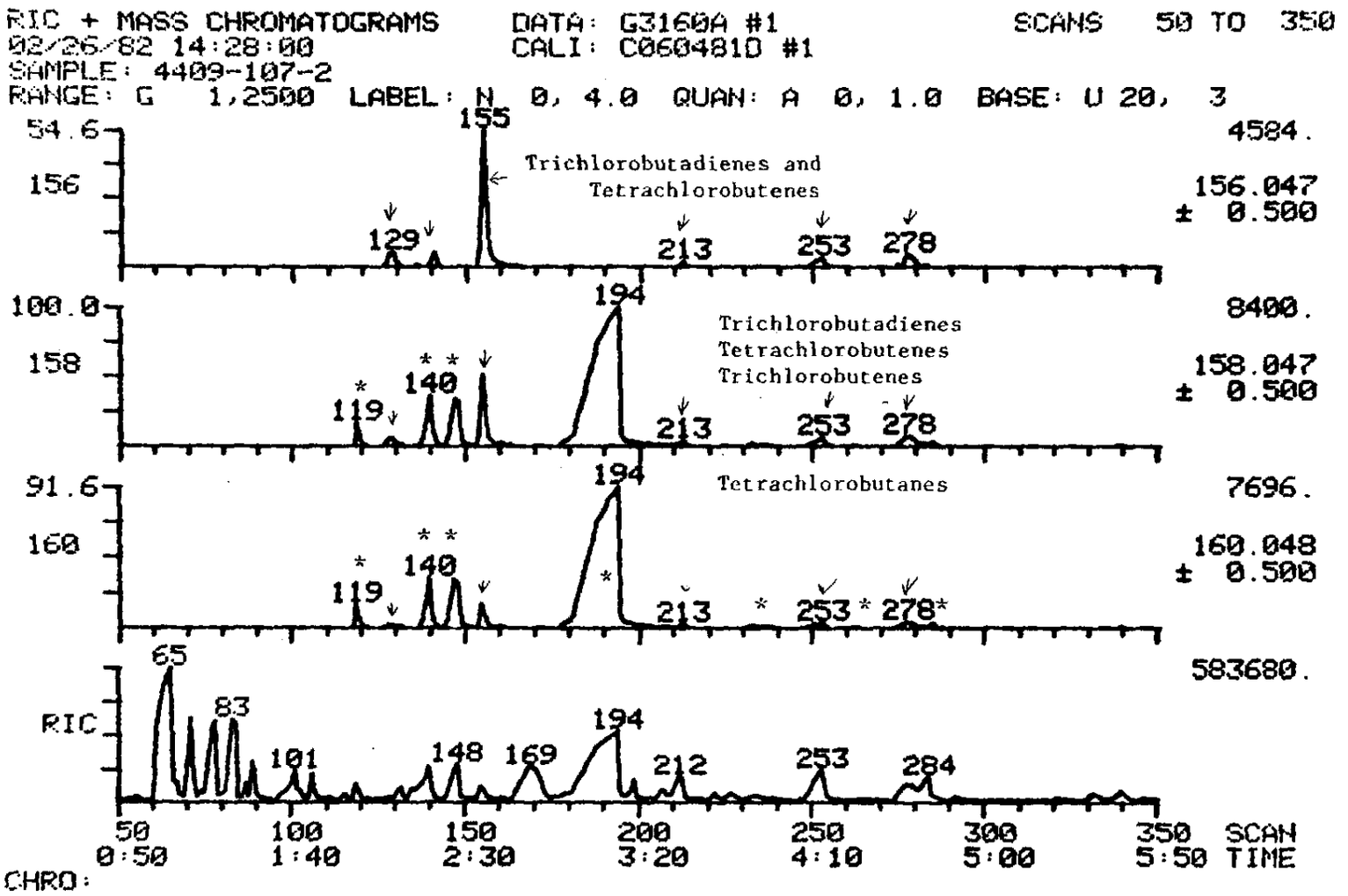

Figure 11. Single Ion Chromatograms of m/e 112, 146, 180 and the Total Ion Chromatogram for Sample No. 4409-107-2

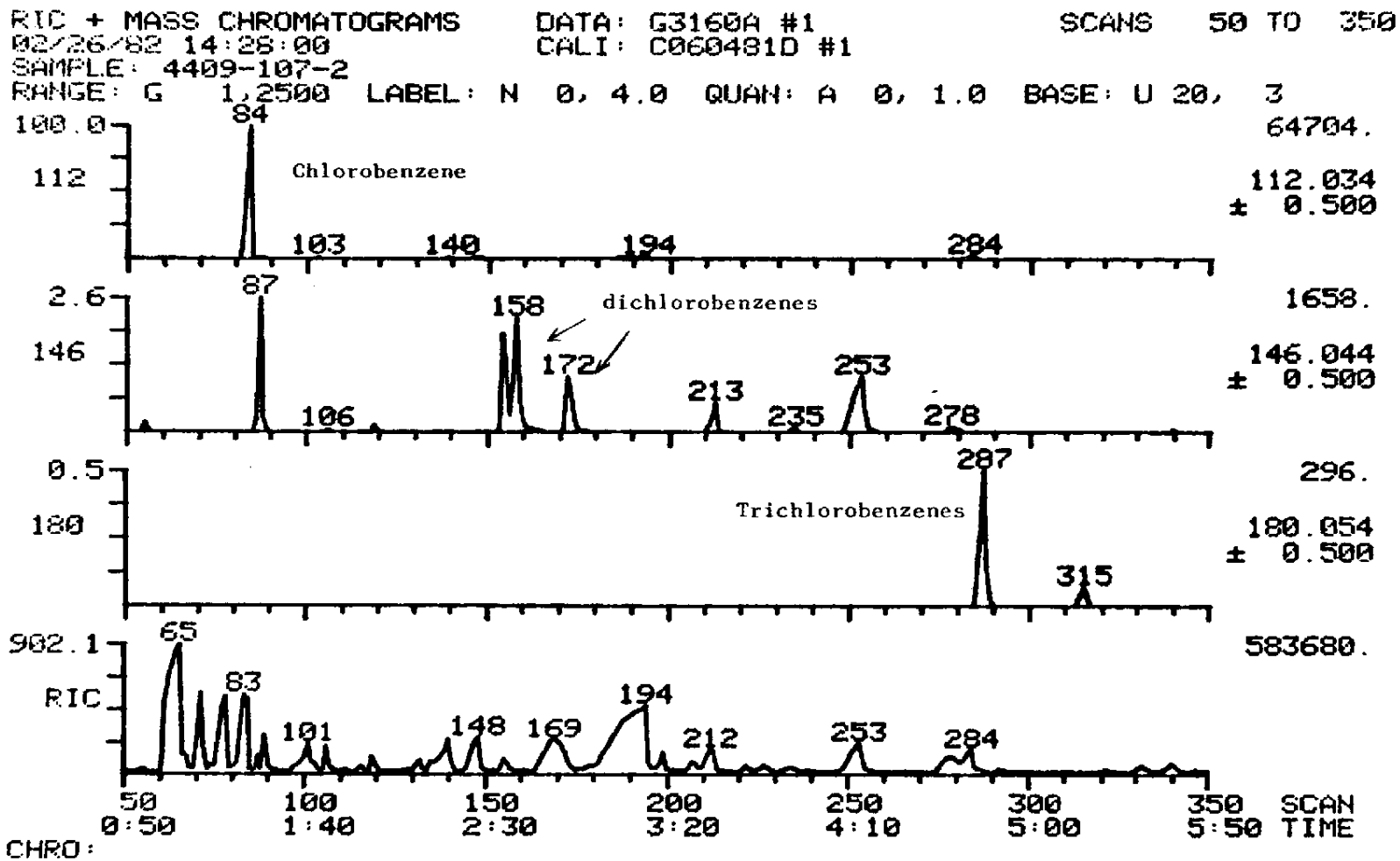

Figure 12. Single Ion Chromatograms of m/e 184, 186, 190, 192 and the Total Ion Chromatograms of Sample No. 4409-107-2

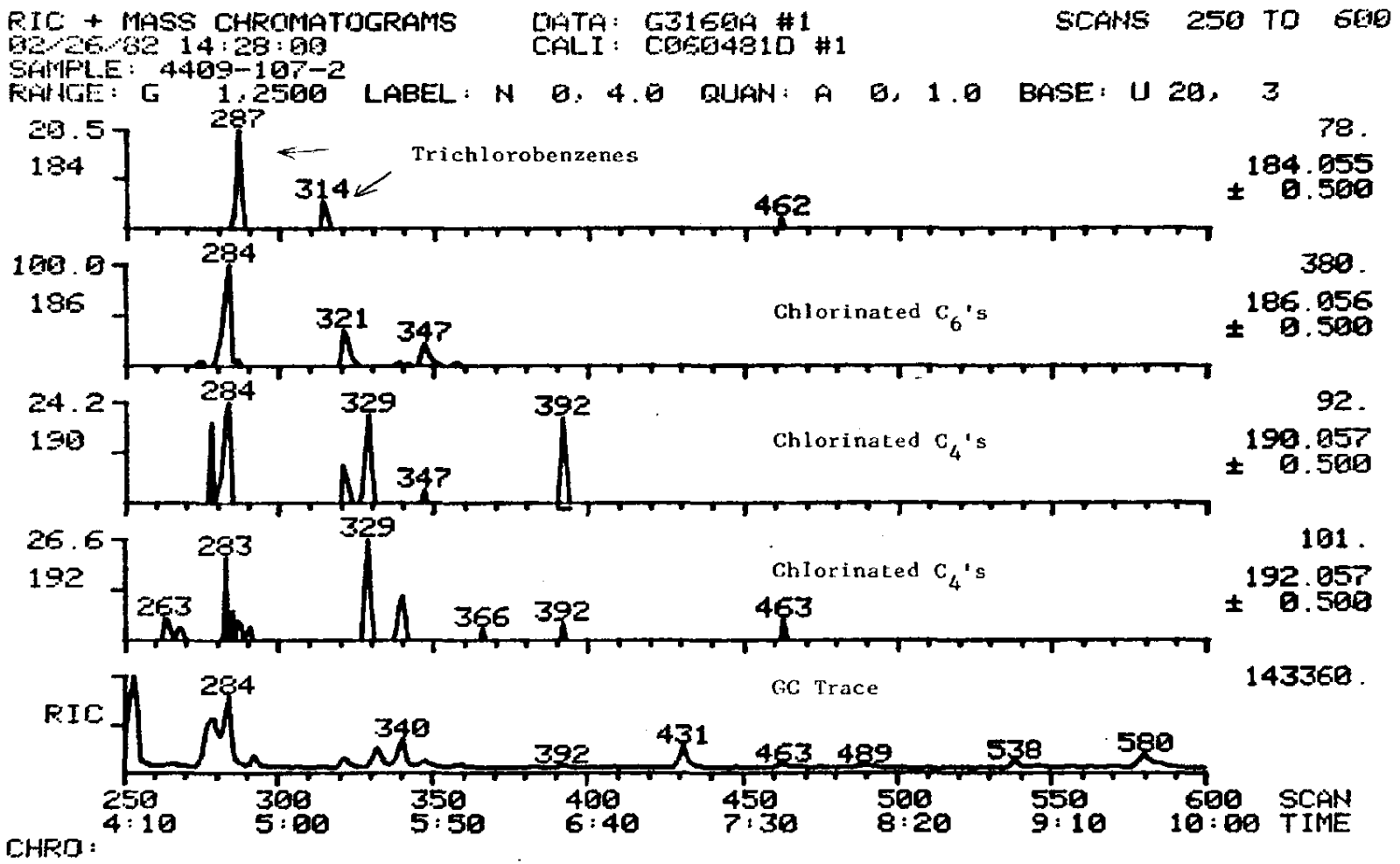

Figure 13. Single Ion Chromatograms of m/e 214, 220, 223, 224 and Total Ion Chromatogram of Sample No.

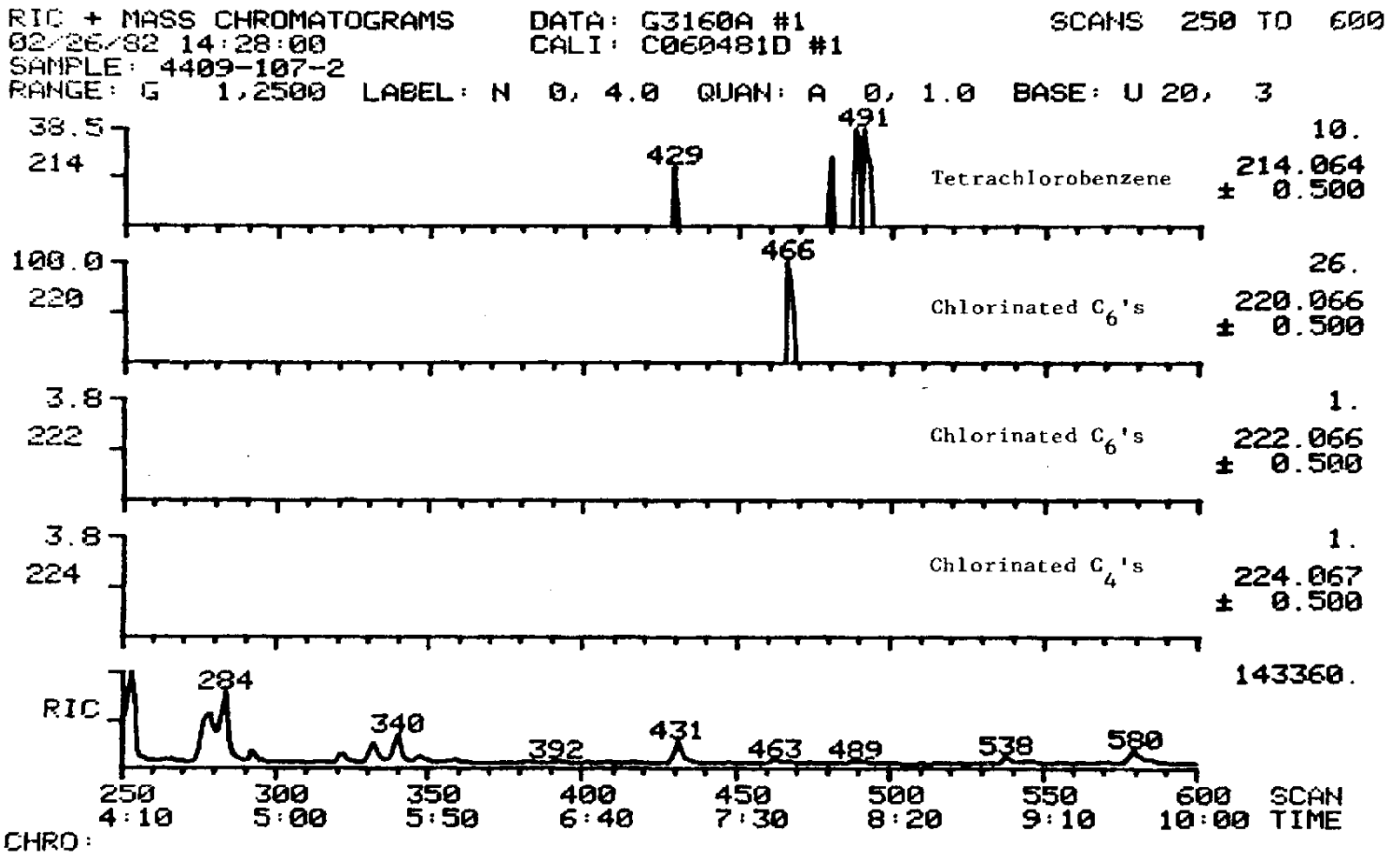

Figure 14. Single Ion Chromatogram for m/e for 207, 209, 244 248, 282 and the Total Ion Chromatogram for Sample No. 4409-107-2