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B.F. GOODRICH COMPANY TECHNICAL REPORT PROJECT REPORT BFG TECHNICAL DOCUMENT

Title Volatiles from a Series of CPVC Samples			PERMANENT RECORD COPY																							
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To: A. J. Olson, R. G. Vielhaber		Location Brecksville R&D Center, D/8546		Project No. 7712-81																						
Summary																										
Volatile compounds from several chlorinated PVC resin samples, including two non-BFG products, were identified by headspace-gas chromatography-mass spectrometry (HS-GC-MS). Some comments are made regarding procedures for quantitative analysis of residuals and water-extractables from CPVC. * * * * * The various CPVC resins listed below were submitted to identify residual volatile compounds: <table border="0"> <tr> <td>MS 1507</td> <td>633 x 561 Lot 2</td> <td rowspan="10"> THE B. F. GOODRICH CO. JUN 04 1981 RESEARCH LIBRARY </td> </tr> <tr> <td>MS 1508</td> <td>Munter's RMS-149</td> </tr> <tr> <td>MS 1509</td> <td>Rhenoflex 22</td> </tr> <tr> <td>MS 1531</td> <td>T-1482</td> </tr> <tr> <td>MS 1532</td> <td>T-382</td> </tr> <tr> <td>MS 1533</td> <td>603 x 560</td> </tr> <tr> <td>MS 1534</td> <td>605 x 560</td> </tr> <tr> <td>MS 1535</td> <td>623 x 563</td> </tr> <tr> <td>MS 1536</td> <td>625 x 563</td> </tr> <tr> <td>MS 1579</td> <td>627 x 563</td> </tr> </table> The initial step was to heat the resin in a sealed, nitrogen-filled vial at ~160°C to enrich the headspace with the low-boiling residuals. A portion of the headspace was withdrawn with a gas syringe and injected into the gas chromatograph to separate the components. Substances eluting from the GC were monitored and identified by mass spectrometry (Varian MAT 311A system). The labeled chromatograms are attached. All samples contained halogenated C₁ and C₂ compounds. Chlorofluoroethane, alkyl benzenes, and other minor components were seen in some samples. The presence of benzene, chlorobenzene and hydrogen chloride in the non-BFG samples may indicate that some decomposition took place upon heating. These decomposition products were not seen in any BFG sample. Experimental conditions for HS-GC: column: 12' x 2 mm ID glass SP2100 program: 40° to 220°C at 5°/min HS: 1-2 mL injection; sample heated at ~160°C for 20-30 min. prior to sampling						MS 1507	633 x 561 Lot 2	THE B. F. GOODRICH CO. JUN 04 1981 RESEARCH LIBRARY	MS 1508	Munter's RMS-149	MS 1509	Rhenoflex 22	MS 1531	T-1482	MS 1532	T-382	MS 1533	603 x 560	MS 1534	605 x 560	MS 1535	623 x 563	MS 1536	625 x 563	MS 1579	627 x 563
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Log Book Reference: SB630, pp 106, 124, 139; MS 1507, 1508, 1509, 1531, 1532, 1533, 1534, 1535, 1536, 1579.																										
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D. E. Ley - Akron			C. H. Lufter - J. B. Pausch																							
R. F. Koebel - E. G. DeCapita - ALTC			P. M. Zakriski																							
C. E. Hoover - Akron, D/3315																										
J. A. Nikora - ALTC																										

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This work was qualitative in nature. Quantitative analysis of residual CHCl_3 and CCl_4 in CPVCs has been performed by solution analysis (DMAC solvent) using gas chromatography with electron capture (EC) detection.¹ An alternate method of analysis that has proven successful is headspace gas chromatography.^{2,3} Headspace GC has several advantages (compared to solution GC) for analysis of polymer residuals; therefore, this technique should be considered for future work on CPVC residuals.

A recent meeting report regarding the analysis of water extractables from CPVC contained the following paragraph:⁴

"In order to analyze for several of USEPA's priority pollutants, the most cost effective way appears to be purchasing a Finnigan GC/MS, according to Zakriski. The going outside lab costs for one sample injected into a Finnigan GC/MS for measurement of 70 compounds is \$1,000. The Finnigan costs \$150,000 installed and can be run by a technician. Because CHCl_3 and CCl_4 are at substantially higher levels in our CPVC pipe compounds than the 139 priority pollutants, quantification of the priority pollutants will not be undertaken at present. If and when we do want to measure such compounds, we should consider buying our own Finnigan."

For the record we wish to clarify some of these statements:

1. The Finnigan GC-MS system is a cost effective way to analyze organics in water⁵, but only in laboratories where the instrument is dedicated to this type of analysis. The quantitative analysis procedures are tedious and time-consuming. Essentially all laboratories that do GC-MS water analysis use one or more instruments full-time for that purpose.
2. The cost of the Finnigan system designed specifically for water analysis (OWA) is ~\$90-100K, not \$150K as stated above.⁴ The "research-grade" Finnigan instruments (4000 series) are more expensive (~\$150 - 200 K).
3. While it is true that the Finnigan OWA system can be "run by a technician,"⁴ any mass spectrometer system needs strong technical and maintenance support to function effectively.⁵ A GC-MS system is a very complex instrument that requires great care and skill in operation.
4. BFGoodrich should consider buying a GC-MS system for water analysis only if (a) there is sufficient work load to utilize the instrument full-time for water analysis, and (b) a qualified technical person is assigned to assume responsibility for operation and maintenance of the system.

A. E. Harris
R. Edgar Harris

R. P. Lattimer
Robert P. Lattimer

References

1. C. E. Hoover, "CPVC Chlorinated Hydrocarbons Testing Update: CHT-UP, Issue 01," April 1, 1981.

2. J. B. Pausch, "Trace Analysis for Carbon Tetrachloride in Geon Hi-Temp Resin Using Headspace Sampling and FID - Gas Chromatography," Project 7719-75, March 14, 1975.
3. J. B. Pausch and I. Sockis, Rubber Chem. Technol., 50, 828(1977).
4. A. J. Olson, "CPVC - Extractables from Potable Water Pipe," Meeting Report, March 10, 1981.
5. R. E. Finnigan, D. W. Hoyt, and D. E. Smith, Environ. Sci. Technol., 13, 534(1979).

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HS-GC-MS, BFG 633 X 561 Lot 2

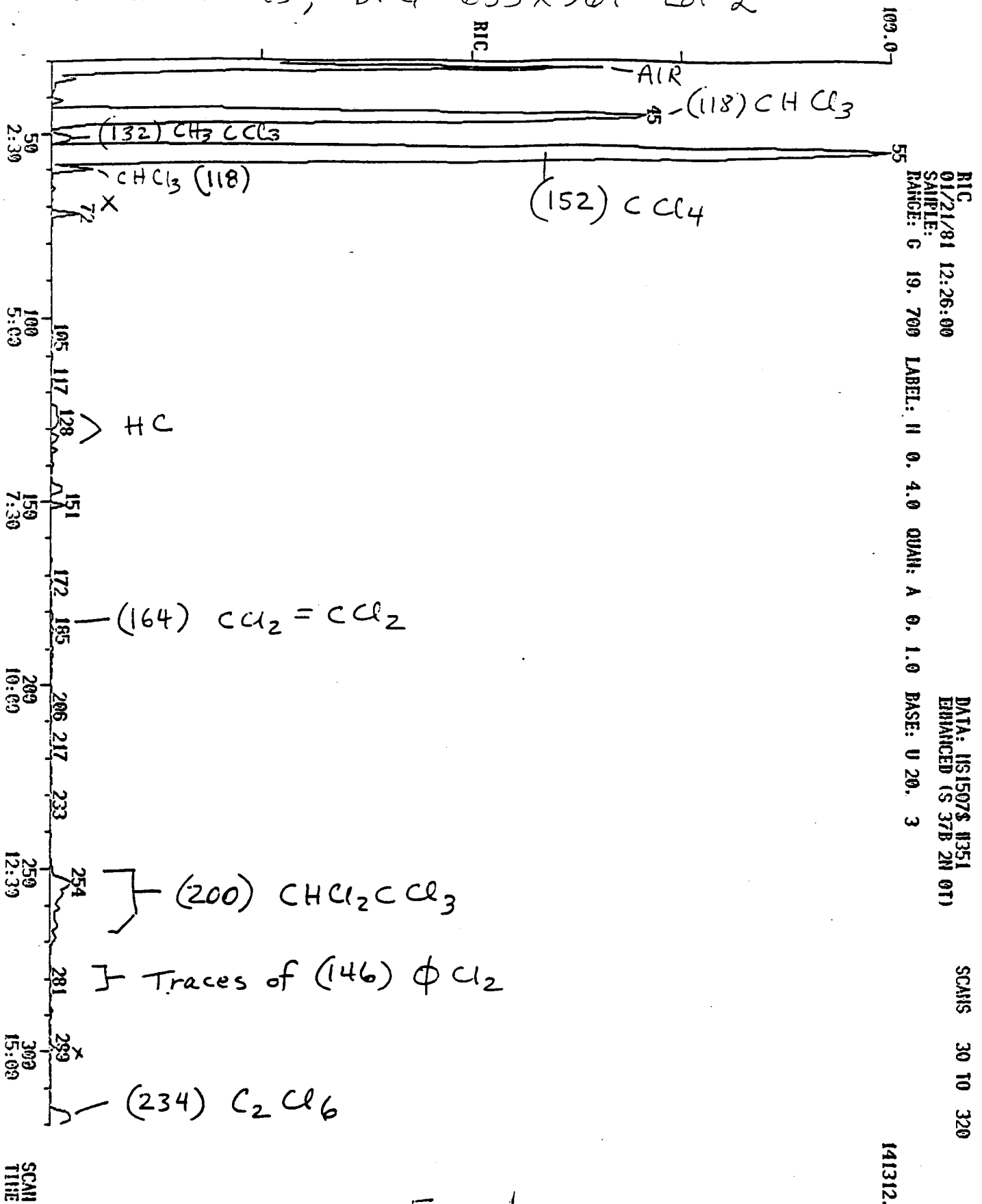
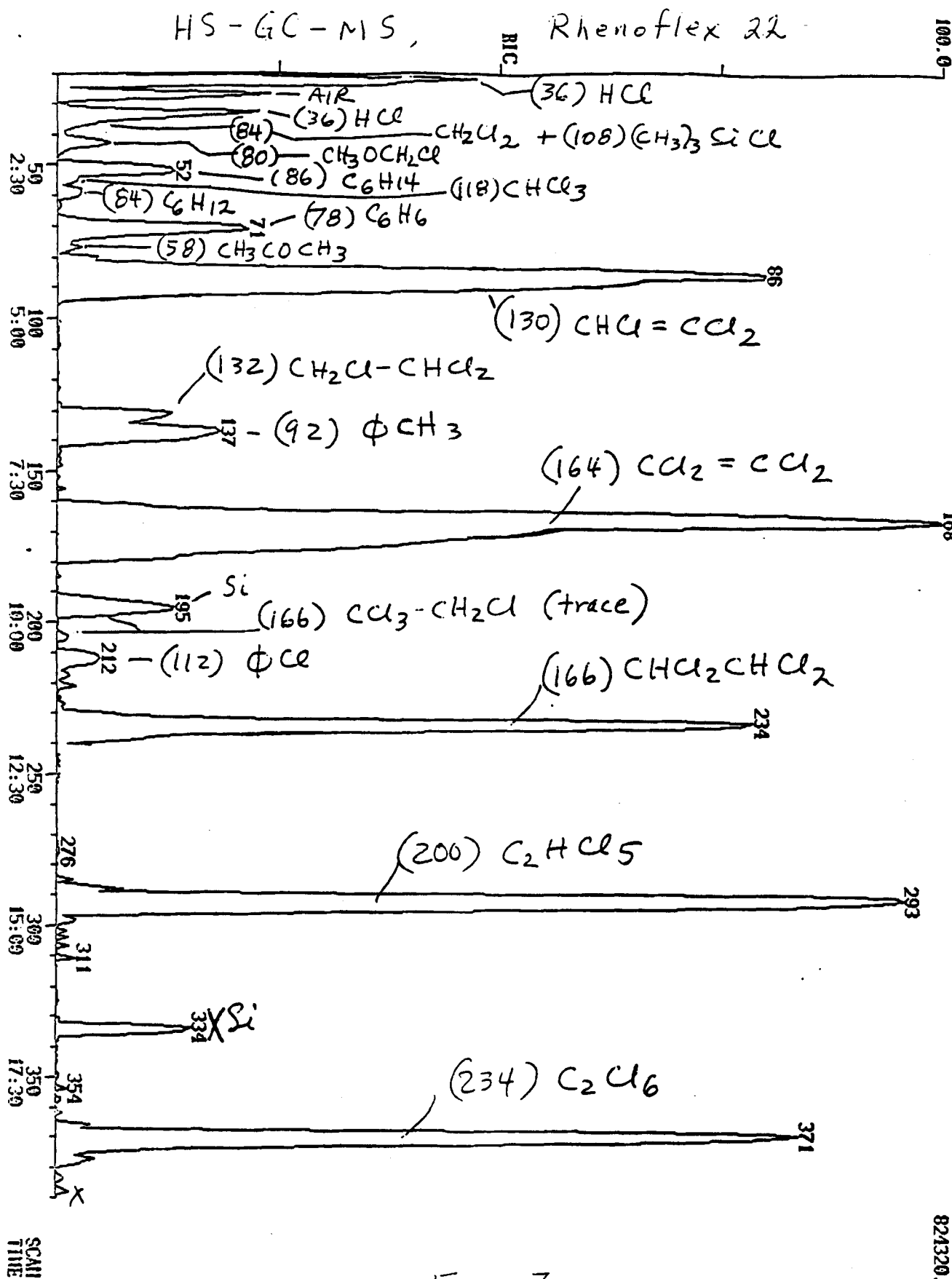


FIG. 1

21124004



RIC
01/21/81 15:30:00
SAMPLE:
RANGE: 6 19.750 LABEL: N 0.40 QUANT: A 0.1.0 BASE: U 20. 3

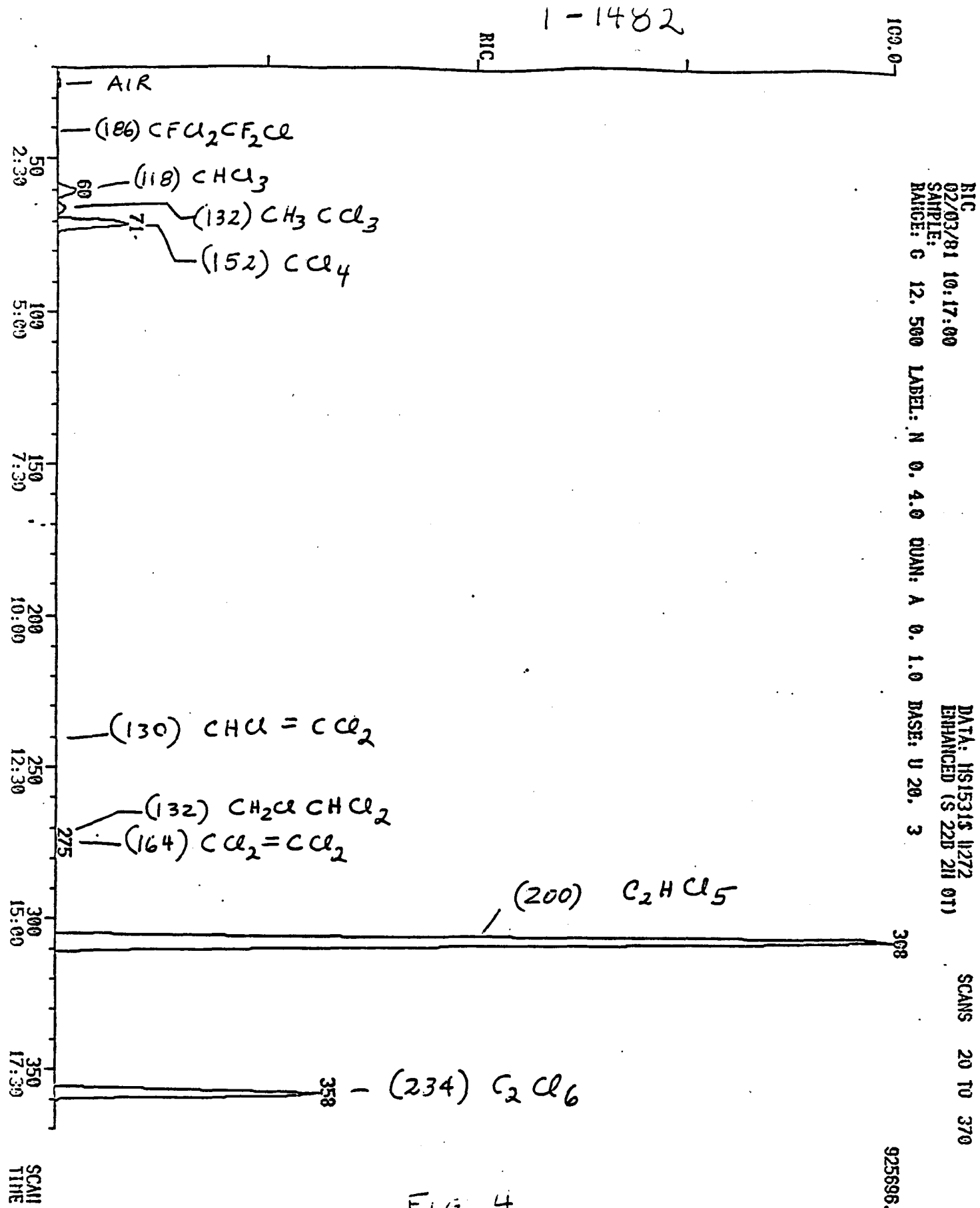
DATA: HS15095.H59
ENHANCED (S 37D 2N 0T)

SCANS 20 10 390

821320.

F. a. 3

21124006



21124007

RIC
02/03/81 12:33:00
SAMPLE:
RANGE: G 12. 500

DATA: HS15328 H466
ENHANCED (S 22B 2N 0T)
LABEL: N 0. 4.0 QUAN: A 0. 1.0 BASE: U 20. 3

SCANS 20 70 400

100.0

T-382

RIC

AIR
(64) 50₂
(186) CFC₂CF₂Cl₂
(118) CHCl₃
(132) CH₃CCl₃
(152) CCl₄

— (164) CCl₂ = CCl₂

(200) C₂HCl₅

(234) C₂Cl₆

391

311

2:30 5:00 7:30 10:00 12:30 15:00 17:30 20:00
SCAN TIME

21124008

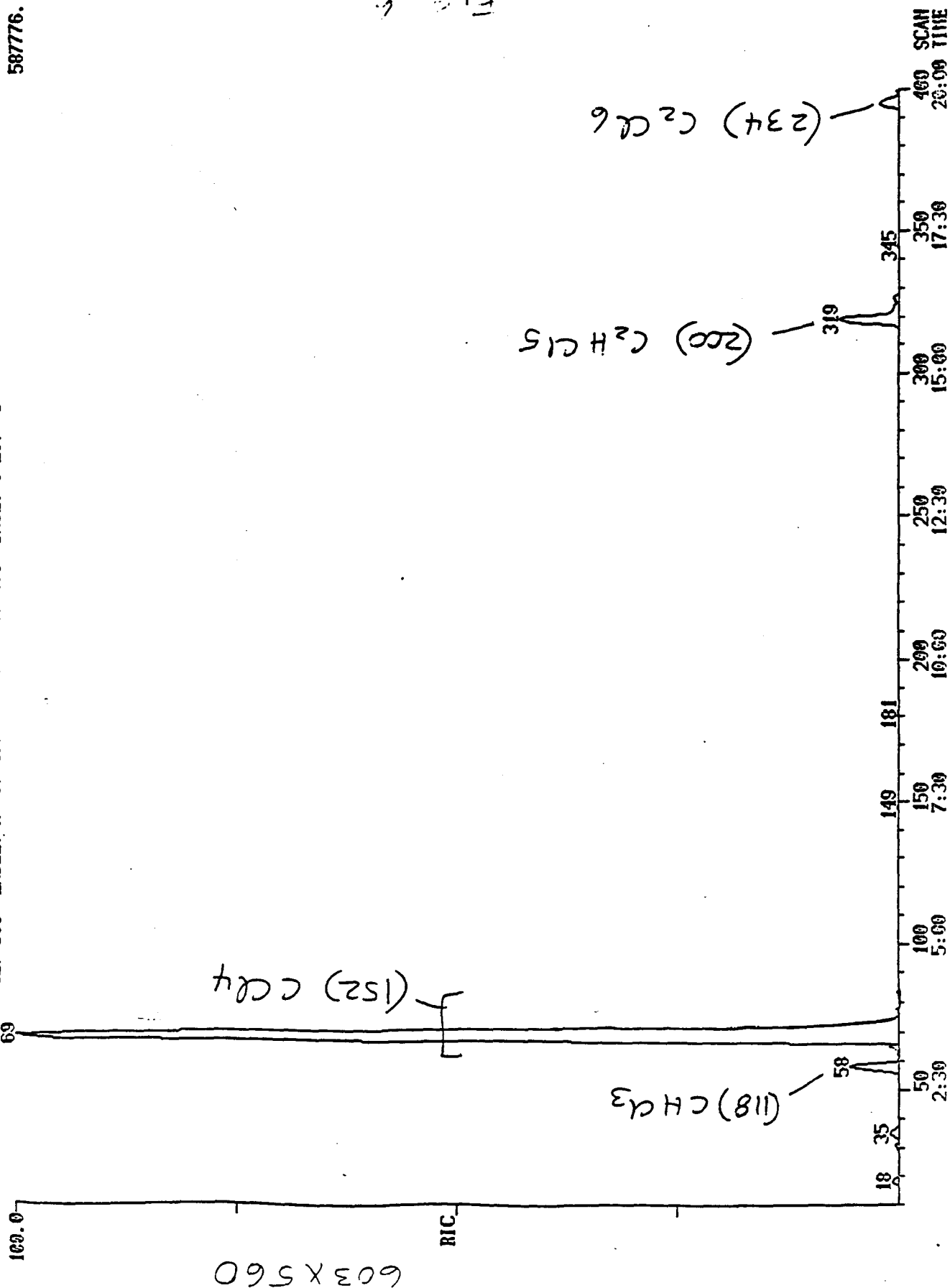
Fig. 5

1292289.

RIC
 02/03/81 13:57:00
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 ENHANCED (S 22B 2N 0T)
 SAMPLE: G 12.500 LABEL: N 0.4.0 QUAN: A 0.1.0 BASE: U 20. 3
 RANGE: G 12.500

SCANS 10 TO 400

587776.



21124009

Fig. 6

RIC
02/03/81 15:05:00
SAMPLE:
RANGE: G 12. 450

DATA: HS1534S #1
ENHANCED (S 22B 2N 01)

LABEL: N 0. 4.0 QUAN: A 0. 1.0 BASE: U 20. 3
311

SCANS 1 TO 450

100.0

605 X 560

RIC

21124010

Fig 7

SCAN
TIME

400
20:00

300
15:00

200
10:00

100
5:00

(152) CCl₄

(118) CHCl₃

(96) C₂H₂Cl₂

(132) CH₃Cl₃

(62) VCM

?

(240) CCl₂Br₂ (?)

(130) C₂HCl₃

-(164) CCl₂ = CCl₂

(200) C₂HCl₅

300
-(234) CCl₆

RIC 02/03/81 15:53:00 DATA: HS1535\$ 01
 SAMPLE: ENHANCED (S 226 2N 0T)
 RANGE: C 12.490 LABEL: H 0.4.0 QUAN: A 0.1.0 BASE: U 20. 3
 59
 395264.

SCANS 1 TO 490

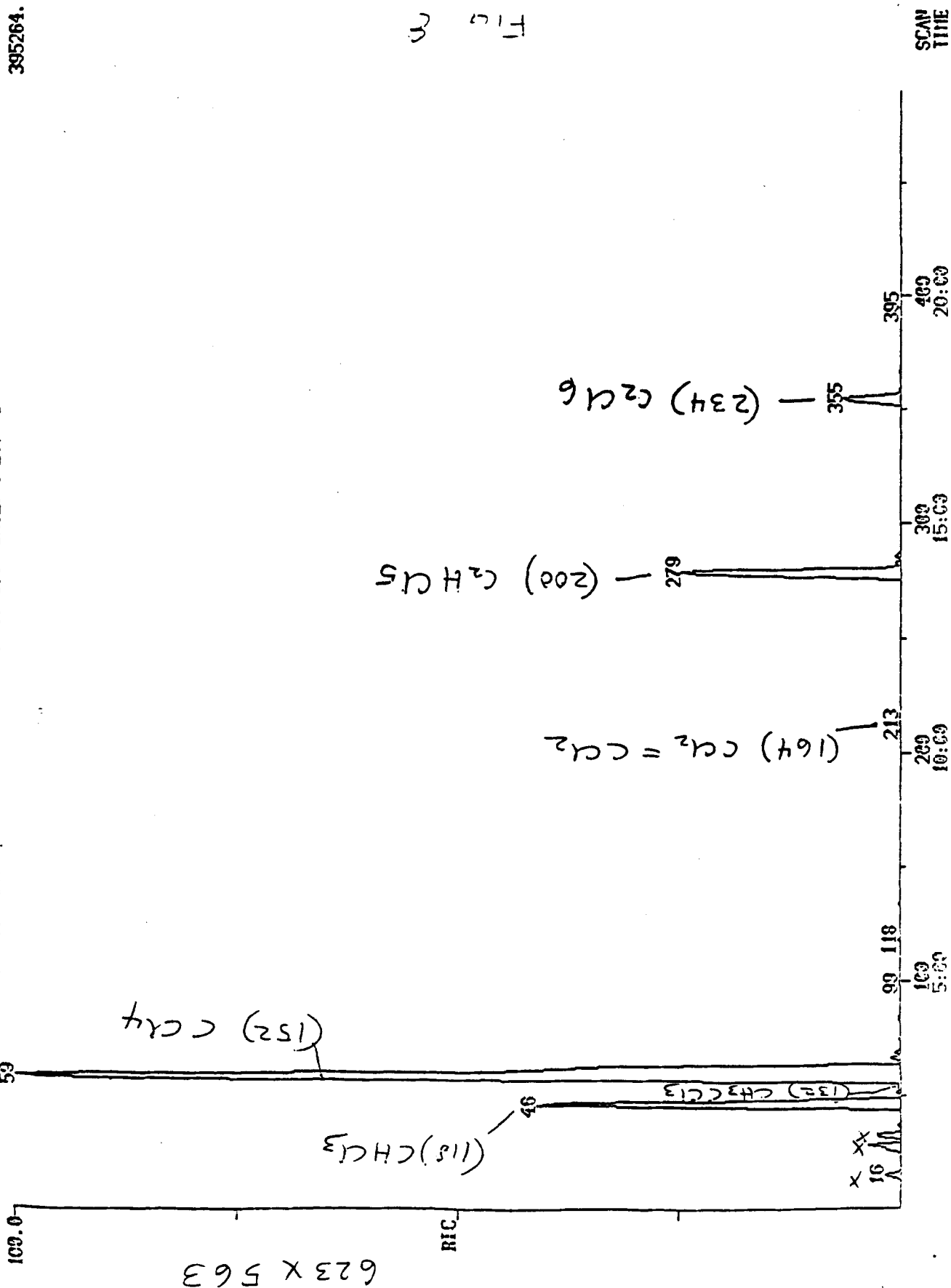
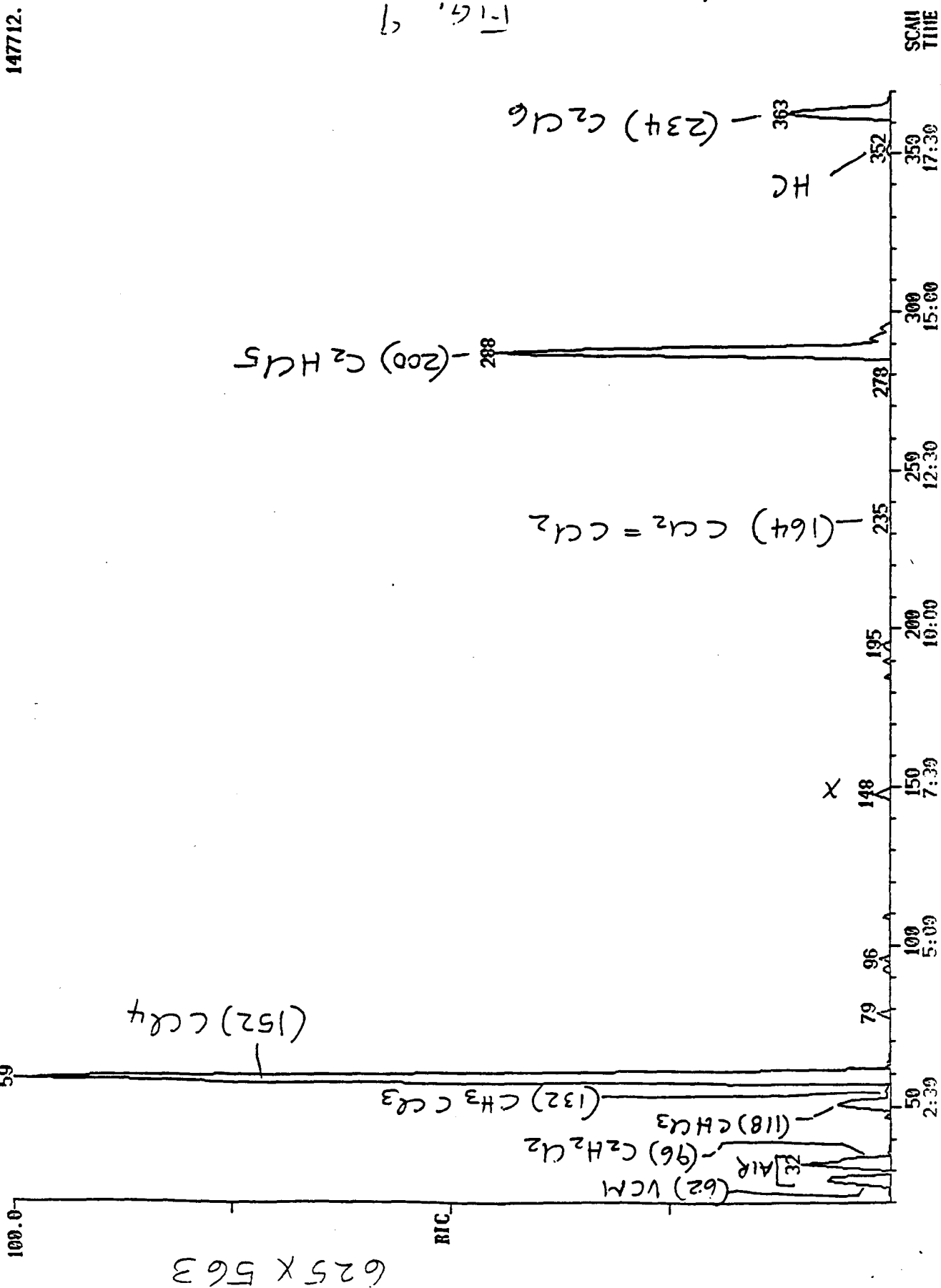


Fig 2

21124011

RIC 02/03/81 16:27:00
 DATA: HS1536\$ B430
 ENHANCED (S 22B 2H 0T)
 SAMPLE: 59
 RANGE: C 12.500 LABEL: N 0.4.0 QUAN: A 0.1.0 BASE: U 20. 3
 SCAHS 20 TO 370
 147712.



627 x 563

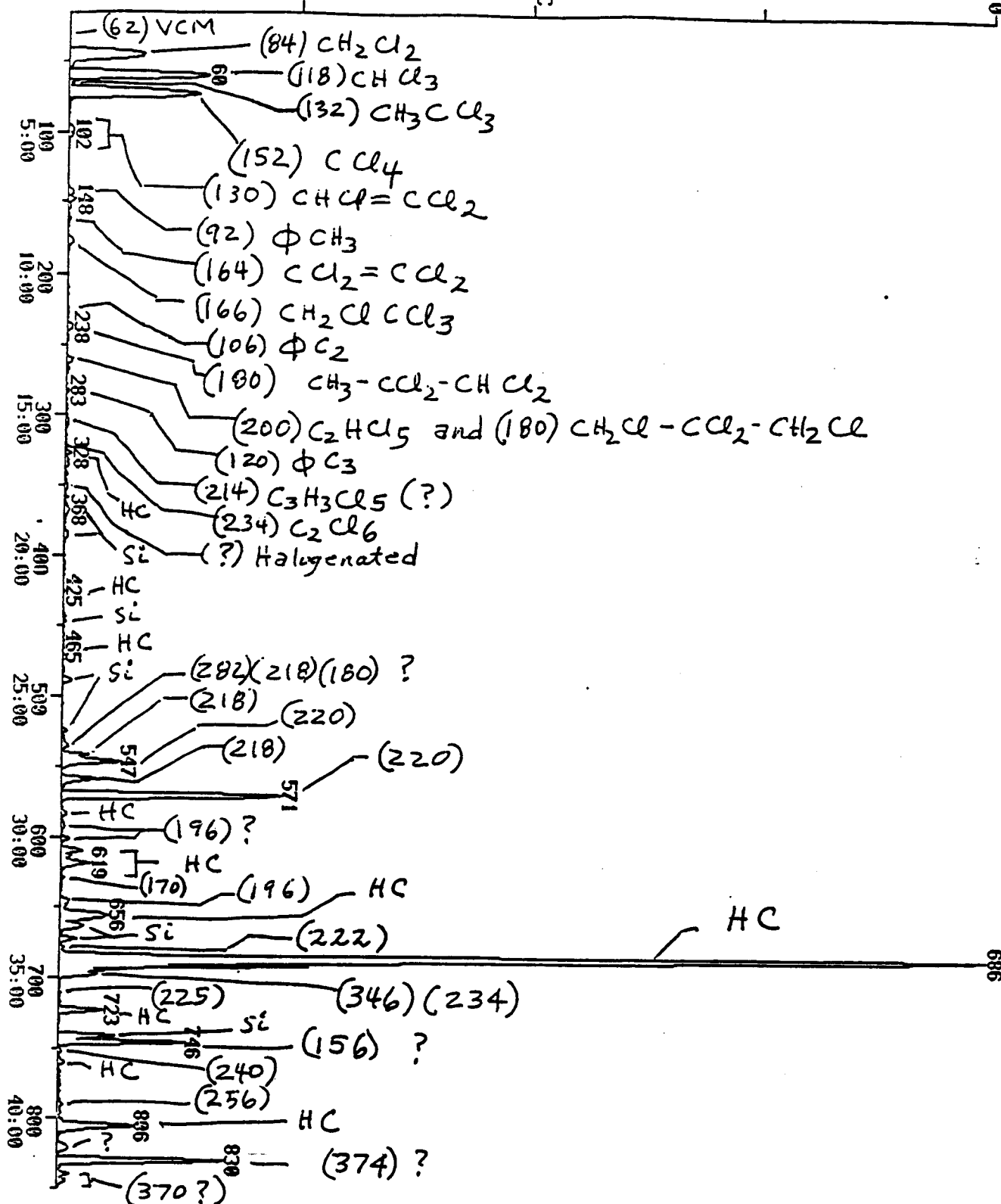
100.0

RIC
02/13/81 10:36:00
SAMPLE:
RANGE: 6 15, 920

DATA: HS1579S #870
ENHANCED (S 298 2N 01)
LABEL: N 0.4.0 QUAN: A 0.1.0 BASE: U 20. 3

SCANS 15 TO 850

532480.



21124013

Fig. 1C