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POLYFUNCTIONALISATION OF CAGE HYDROCARBONS (II)
ERLANGEN-NÜRNBERG UNIV (GERMANY) P. B. INST. FÜR
ORGANISCHE CHEMIE P. VON RAGUE SCHLEYER MAR 88

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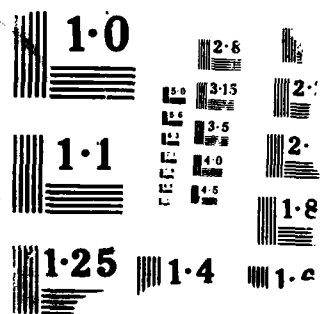
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POLYFUNCTIONALISATION OF CAGE HYDROCARBONS

Second Interim Report

by

Prof. Dr. Paul von Rague Schleyer

March 1988

United States Army
European Research Office of the U.S. Army
London, England.

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INTRODUCTION.

This report outlines the work being carried at the University of Erlangen-Nurnberg aimed at the synthesis of polyhalogenated cage molecules, their isolation, characterisation, and assessment as potential pre-cursors to "high density" materials.

The progress in this field up to 31/3/88 is reported herein and intended future work is described.

The "cage" systems studied are adamantane ($C_{10}H_{16}$), diamantane ($C_{14}H_{20}$), and norbornadiene-dimer ($C_{14}H_{16}$) (NBD).

CONTENTS.

- (I) Photobromination of 1,3,5,7-tetrabromoadamantane ($AdBr_4$).
- (II) Photochlorination of $AdBr_4$.
- (III) Photochlorination of adamantane and 1,3,5,7-tetrachloroadamantane. ($AdCl_4$).
- (IV) Chlorination of diamantane.
- (V) Chlorination of NBD.
- (VI) Ionic bromination of diamantane.
- (VII) Conclusions and intended further work.

(I) Bromination of $AdBr_4$.

A variety of different methods have been employed all without success, the $AdBr_4$ remaining unreacted. The methods attempted are shown in the scheme below.

- (1a) $AdBr_4 + Br_2 + CCl_4 \xrightarrow{h\nu, RT}$ no reaction
- (1b) As above + $N=NC(CH_3)_2CN \xrightarrow{h\nu, RT}$ no reaction
- (2) $AdBr_4 + Br_2 \xrightarrow{h\nu}$ no reaction
- (3) $AdBr_4 + NBS + \text{dibenzoylperoxide} \xrightarrow{\text{heat}}$ no reaction
- (4) $AdBr_4 + Br_2 + HgO \xrightarrow{CCl_4, \text{heat}}$ no reaction
- (5) $AdBr_4 + Br_2 + HgO \xrightarrow{BrCl_3, C}$ no reaction

(II) Photochlorination of $AdBr_4$.

On short term photochlorination, typically 30-60 mins., a total halogen content of 8 or 9 is achieved.

- (1) $AdBr_4 \xrightarrow{Cl_2, h\nu} AdBr_4Cl_4 + AdBr_4Cl_5$

On longer photolysis, up to 3 days, a higher halogen content is obtained. This product is not yet fully analysed but from 1H NMR is estimated to contain a total halogen content of around 12.

- (2) $AdBr_4 \xrightarrow{Cl_2, h\nu} AdBr_4Cl_7$
 1H nmr(m.4.0, m.6.1ppm)

(III) Photochlorination of Adamantane.

20hrs. photochlorination results, after work up, in a product mixture of the following composition:

$C_{10}Cl_{12}H_4$ 68%, $C_{10}Cl_{13}H_3$ 29%, $C_{10}Cl_{14}H_2$ 3%.

This composition is calculated by comparing the peak intensities for the $(M-Cl)^+$ ions. (The M^+ peaks are very weak for these compounds). The mixture exhibits the 1H nmr spectrum; (m,3.65, m,6.0ppm)

Chlorination of Tetrachloroadamantane $AdCl_4$.

Short term photolysis results in the formation of $AdCl_6$ and $AdCl_8$ which together exhibit the 1H nmr spectrum (m,3.3, m,5.2ppm).

Photolysis for 52hrs results in further chlorination, the product found by mass spec. to be a mixture of $Cl_{11}, Cl_{12}, Cl_{13}$ and Cl_{14} compounds. This mixture exhibits the 1H nmr spectrum (m,3.6 CH_2 , m,5.8 $CHCl$).

Partial evaporation of a solution of this mixture in Et_2O results in crystallisation. X-Ray analysis of these crystals reveals that they belong to the Cubic system but also that they are severely disordered indicating the probability that the very similar packing properties of the $Cl_{11}-Cl_{14}$ compounds allow them to crystallise together. Further X-Ray analysis on these crystals was therefore impossible due to the disorder. Mass spec. analysis of these crystals indicates the following approximate composition:

$AdCl_{12}H_4$ 49%, $AdCl_{13}H_3$ 38%, $AdCl_{14}H_2$ 13%.

The 400MHz 1H nmr spectrum is provided (spec.1)

HPLC separation of this crystalline mixture using petrolether/ Et_2O 95:5 %vol gives two distinct fractions(1 and 3) which were at first thought to be only mixtures enriched in one or other chloroadamantane. It now seems more probable that the two fractions, although exhibiting similar mixture compositions, in fact contain distinct isomers of these compounds.

The compositions of the fractions, estimated by comparison of the intensities of the $(M-Cl)^+$ peaks in the mass spectra, are as follows;

FRACTION 1	COMPOUND	FRACTION 3
39%	AdH_4Cl_{12}	46%
35%	AdH_3Cl_{13}	54%
26%	AdH_2Cl_{14}	--

In both fractions, the compounds of molecular formula AdH_4Cl_{12} and AdH_3Cl_{13} are major components and present in fairly equal amounts. The 400MHz 1H nmr spectra of these two fractions are, however, very different. Spectra provided (spec.2 and spec.3).

The spectrum for fraction 1 (spec.2) exhibits many resonances in the region which corresponds to the R_2CHCl function (~6.0ppm) and also a few less intense peaks in the R_2CH_2 region.

All of these peaks are absent from the spectrum of fraction 3 (spec.3). An explanation of this is that fraction 1 contains mainly those isomers of the Cl_{12}, Cl_{13} and Cl_{14} compounds which contain no R_2CH_2 groups. This is consistent with the fact that these isomers have inherently lower dipoles than those not containing such groups, and therefore would be eluted first in the HPLC by the non-polar solvent.

As mentioned, only fraction 1 contains the $AdCl_{14}H_2$ isomers all of which probably contain no R_2CH_2 groups. Fraction 3, on the other hand, contains those isomers of $AdCl_{12}H_4$ and $AdCl_{13}H_3$ which do contain a R_2CH_2 group. These have fewer isomeric possibilities. This fact and the absence of $AdCl_{14}H_2$ combine to greatly simplify the nmr spectrum (spec.3).

Semi empirical molecular mechanics (MNDO) calculations on the isomers of $AdCl_{12}H_4$ containing no R_2CH_2 groups show the following;

ISOMER	RELATIVE ENERGY Kcal/Mol.
C _s	0.0
S ₄	1.0
C _{2v}	1.9
all C ₁	>6.8

These results clearly show that the C₁ isomers are energetically disfavoured and this agrees with both the ¹H and ¹³C spectra (spec.2 & 4) which do not contain enough peaks for the C₁ isomers to be accounted for.

The C_s, S₄, and C_{2v} isomers could conceivably all be produced although in differing amounts. This also agrees with the spectral data.

More detailed calculations on these isomers and on those containing R₂CH₂ groups could help identify those isomers formed in preference, which can only be decided unambiguously by X-Ray analysis, which, as mentioned earlier, poses problems in crystal growth due to the similarity in the packing characteristics of these spherically shaped molecules.

(IV) Chlorination of diamantane.

Ionic chlorination of diamantane using AlCl₃ results in the formation of C₁₄H₁₆Cl₄ which is highly insoluble in anything except THF.

Photochlorination of diamantane, for approximately 24hrs results in a mixture of the following estimated composition (mass spec.).

C₁₄H₁₀Cl₁₀ 45%, C₁₄H₉Cl₁₁ 45%, C₁₄H₈Cl₁₂ 9%, C₁₄H₇Cl₁₃ 1%.

(V) Chlorination of norbornadiene dimer (NBD) (C₁₄H₁₆)

Ionic chlorination of NBD results in products in the Cl₄-Cl₇ range. The composition estimated from the mass spectrum is as follows;

C₁₄H₁₂Cl₄ 35%, C₁₄H₁₁Cl₅ 56%, C₁₄H₁₀Cl₆ 9%.

Photochlorination of NBD, proceeds somewhat further yielding compounds in the Cl₈-Cl₁₀ range. Composition estimate from mass spec. is as follows;

C₁₄H₈Cl₈ 6.5%, C₁₄H₇Cl₉ 81%, C₁₄H₆Cl₁₀ 12.5%.

The Cl₉ compound precipitates from the reaction. The Cl₈ compound can be obtained in 92% purity by filtering off the Cl₉ compound and working up.

(VI) Bromination of diamantane.

A selective method for the synthesis of 4,9-dibromodiamantane from diamantane in 85% yield has been developed.



This compound may prove to be a more suitable starting point than tetrabromodiamantane, for further halogenation, due to its higher solubility in the solvents usually employed in photo-halogenations.

(x) Conclusions and intended future work.

Chlorination of adamantane has now succeeded in producing major products in the Cl_{12} - Cl_{14} range which corresponds to over 75% functionalisation.

Partial separation of the isomers produced has been achieved by HPLC in quantities which allow analysis by 400MHz ^1H nmr.

It has not yet been possible to separate the Cl_{12} and Cl_{13} compounds by this method but separation of the isomers of these compounds which contain unfunctionalised R_2CH_2 groups from those which do not, has been achieved. Further identification requires larger quantities of separated isomers for ^{13}C and ^{13}C - ^1H shift correlated spectroscopy.

Reversed phase HPLC could be useful in separating AdCl_{12} and AdCl_{13} from pre-separated R_2CH_2 containing, and non-containing isomer mixtures.

Further MNDO calculations on the various isomers of AdCl_{12} , AdCl_{13} , and AdCl_{14} will aid identification of preferentially formed isomers. The growth of crystals suitable for X-Ray analysis is being actively pursued.

Photochlorination of AdCl_4 also proceeds to the Cl_{12} - Cl_{14} range. It is not yet clear whether the isomeric distribution here is the same as it is in the case of directly photochlorinated adamantane. Again samples are being purified for ^{13}C and ^{13}C - ^1H correlated nmr spectroscopy.

Photobromination has been somewhat less successful, with the attempted bromination of pre-halogenated adamantanes not proceeding at all, and that of adamantane only to the Br_3 - Br_5 range.

Chlorination of other cage systems, diamantane, and NBD has also proceeded well, products in the Cl_{12} - Cl_{14} and Cl_7 - Cl_9 ranges being produced respectively. This corresponds to 70% and 50% functionalisation.

Photochlorination of ionically brominated cages has begun with initial experiments yielding AdBr_4Cl_4 - AdBr_4Cl_6 compounds which appear to be more amenable as far as crystallisation is concerned.

Two computer programs have been written which significantly simplify the interpretation of the mass spectra of these highly halogenated and mixed halogen compounds.

Methods of halogen exchange (from Cl or Br to I) are being investigated with some success.

Scientific personnel involved in this work:

Prof. Dr. P.v.R. Schleyer (Principal Investigator)



Dr. Peter Gregory (post doctoral associate)

Erlangen, April 6, 1988

1,0,4,5,6,7-TETRACHLOROADAMANTINE

SPECTRUM 1

OBS ^1H 400 MHz

PRE - HPLC

$\text{C}_{10}\text{H}_4\text{Cl}_{12}$ 49%

$\text{C}_{10}\text{H}_3\text{Cl}_{13}$ 38%

$\text{C}_{10}\text{H}_2\text{Cl}_{14}$ 13%

MASS SPEC.

ESTIMATE

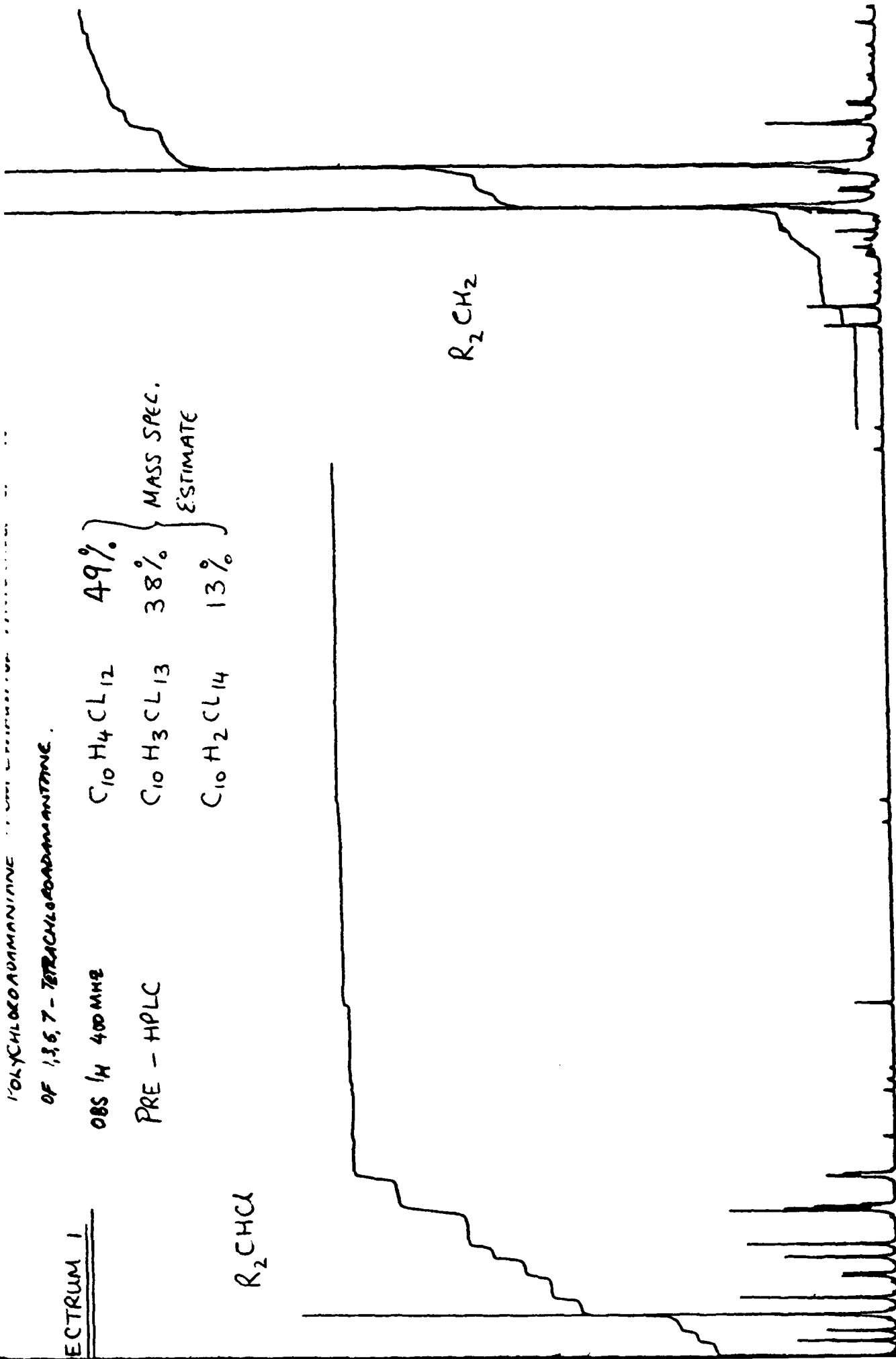
R_2CHCl

R_2CH_2

5 ppm

4

6



RESOL 0.3100000 Hz
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 ABOBS 399724.80000000 MHz
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 COMPT 85095/P. GREGORY/CL-ADAPTANTANE/CDCL3/1H/MS/+24DEG

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1	7.52210	0.07589	3007.20	5893	
2	7.26332	6.64536	2903.75	6232	
3	6.28699	0.18926	2513.63	7511	
4	6.22668	0.07961	2689.32	7590	
5	6.21523	0.23879	2484.74	7605	
6	6.18867	1.07743	2466.13	7666	
7	6.13584	0.15178	2453.00	7709	
8	6.11600	0.24379	2445.07	7735	
9	6.10378	0.07267	2440.19	7751	
10	6.09386	0.09826	2436.22	7764	
11	6.08775	0.09212	2433.78	7772	
12	6.05364	0.11009	2420.96	7814	
13	6.04300	0.71671	2416.69	7828	
14	6.04195	0.22613	2415.47	7832	
15	6.03090	7.66135	2410.89	7847	
16	6.02363	0.21194	2408.14	7856	
17	6.01905	0.12724	2406.31	7862	
18	6.01371	0.08142	2404.17	7869	
19	5.99539	1.50144	2396.85	7893	
20	5.97401	1.07475	2388.31	7921	
21	5.95188	0.18569	2379.46	7950	
22	5.94043	8.83748	2374.88	7965	
23	5.92898	0.15134	2370.30	7980	
24	5.90684	2.32709	2361.45	8009	
25	5.87325	0.11562	2343.22	8053	
26	5.86256	0.84685	2343.75	8067	
27	5.85722	0.83090	2341.61	8074	
28	5.82287	1.66055	2327.88	8119	
29	5.80836	0.07892	2322.08	8138	
30	5.79615	2.21793	2317.20	8154	
31	5.73966	0.10553	2294.62	8228	
32	5.72745	2.46002	2289.73	8244	
33	5.72287	1.65931	2287.90	8250	
34	5.71905	1.22502	2286.38	8255	
35	5.71447	0.50939	2284.55	8261	
36	5.65722	1.03668	2261.66	8336	
37	5.65340	0.64309	2260.13	8341	
38	5.65188	0.79604	2259.52	8343	
39	5.64348	0.08886	2256.16	8354	
40	5.57707	0.18735	2229.61	8441	
41	5.48241	0.17256	2191.77	8565	
42	5.46180	0.09908	2183.53	8592	
43	5.45951	0.12049	2182.62	8595	
44	5.43585	0.07760	2173.16	8626	
45	5.30150	0.56185	2119.45	8802	
46	4.93051	0.12530	1971.13	9286	
47	4.88547	0.15218	1953.13	9347	
48	4.16486	0.15409	1665.04	10291	
49	4.11982	0.12804	1647.03	10350	
50	3.90609	0.85329	1561.58	10630	
51	3.86715	1.09685	1546.02	10681	
52	3.83891	0.11375	1534.73	10718	
53	3.80227	0.10272	1520.08	10766	
54	3.76181	0.20836	1503.91	10819	
55	3.75952	0.14256	1502.99	10822	
56	3.75494	0.19437	1501.16	10828	
57	3.75112	0.14359	1499.63	10833	
58	3.74502	0.38807	1497.19	10841	
59	3.73891	0.15056	1494.75	10849	
60	3.72822	0.23019	1490.48	10863	
61	3.71067	0.65117	1483.46	10886	
62	3.68853	0.10376	1474.61	10915	
63	3.68242	0.08788	1472.17	10923	
64	3.67174	0.92214	1467.90	10937	
65	3.66563	1.33818	1465.45	10945	
66	3.65952	15.65355	1463.01	10953	
67	3.65189	0.86309	1459.96	10963	
68	3.62746	0.60119	1450.20	10995	
69	3.62135	0.56634	1447.75	11003	
70	3.61143	0.10793	1443.79	11016	
71	3.59158	0.54759	1435.85	11042	
72	3.58929	0.90133	1434.94	11045	
73	3.58700	0.65789	1434.02	11048	
74	3.57555	16.54562	1429.44	11063	
75	3.56868	0.93355	1426.70	11072	
76	3.54426	0.14219	1416.93	11104	
77	3.53891	0.12695	1414.79	11111	
78	3.52288	0.16527	1405.39	11132	
79	3.51754	0.08055	1406.25	11139	
80	3.49006	1.63376	1395.26	11175	
81	3.48471	0.68254	1393.13	11182	
82	3.47555	0.17007	1389.47	11194	
83	3.45189	0.43288	1380.00	11225	
84	3.44685	0.46387	1377.87	11232	
85	3.44044	0.16844	1375.43	11240	
86	3.40181	0.13010	1359.86	11291	
87	3.39235	0.14425	1354.20	11303	
88	3.36868	0.09383	1348.74	11334	
89	3.36478	0.09447	1348.83	11337	

CHCl₃

R₂CHCl

R₂CH₂

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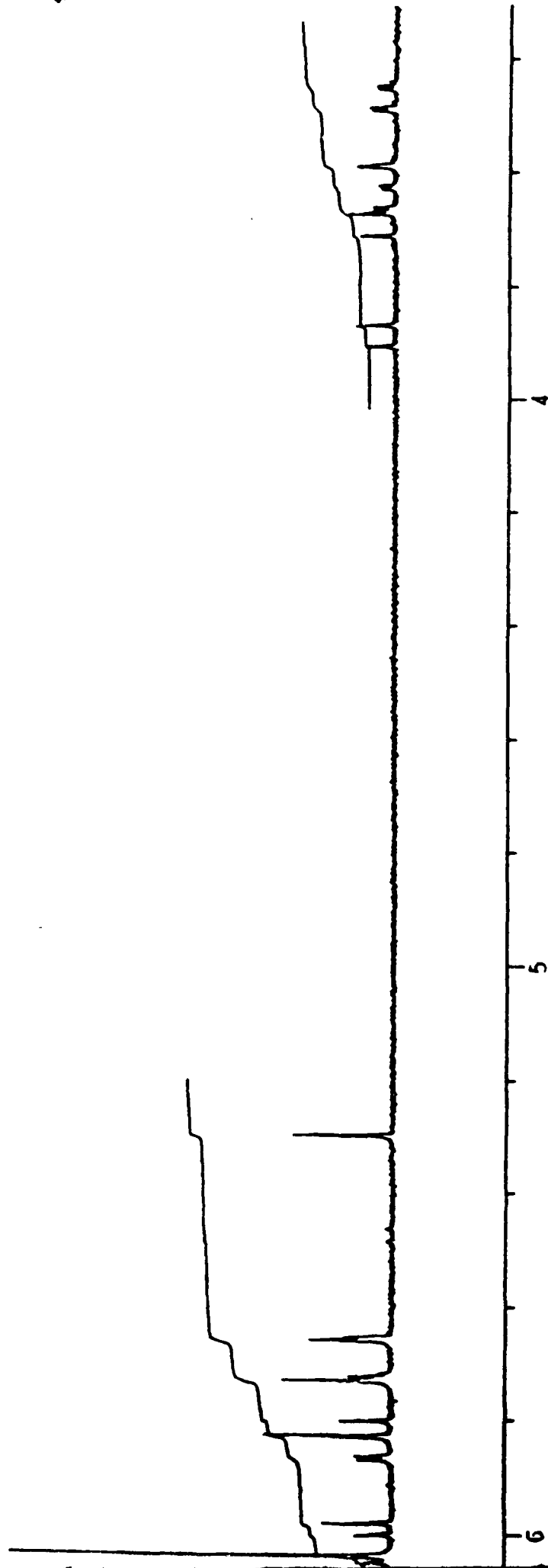
OBS ^1H 400 MHz

SPECTRUM 2

$\text{C}_{10}\text{H}_4\text{Cl}_{12}$	39%	MASS SPEC ESTIMATE
$\text{C}_{10}\text{H}_3\text{Cl}_{13}$	35%	
$\text{C}_{10}\text{H}_2\text{Cl}_{14}$	26%	

R_2CHCl

R_2CH_2



14-DEC-87 11:28:24

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NO.	PPM	INT(%)	FREQ(Hz)	POSITION	BAR GRAPH
1	7.26561	5.49368	2904.66	6231	+ CHCl ₃
2	6.17630	0.66949	2469.18	7658	+ R ₂ CHCl
3	6.05111	0.68401	2419.13	7822	
4	6.03813	6.27609	2413.94	7839	
5	6.00226	0.69371	2399.60	7886	+ R ₂ CH ₂
6	5.98165	1.22259	2391.36	7913	
7	5.87020	0.65814	2346.80	8059	
8	5.86409	0.69479	2344.36	8067	
9	5.82898	2.15283	2330.32	8113	
10	5.80302	0.94313	2319.95	8147	
11	5.73432	1.85953	2292.48	8237	
12	5.72669	0.79241	2289.43	8247	
13	5.66333	1.43001	2264.10	8330	
14	5.65875	0.89304	2262.27	8336	
15	5.30302	1.71058	2120.06	8802	
16	3.90990	0.57573	1563.11	10627	
17	3.87173	0.73602	1547.85	10677	
18	3.71372	0.65704	1484.68	10884	
19	3.67555	0.85458	1469.42	10934	+ R ₂ CH ₂
20	3.67097	0.46937	1467.59	10940	
21	3.66487	0.45714	1465.15	10948	
22	3.59235	0.72266	1436.16	11043	
23	3.49464	0.47613	1397.09	11171	
24	3.48929	0.51757	1394.96	11178	
25	2.34045	0.55242	935.67	12683	
26	2.33052	0.53226	931.70	12696	
27	2.21449	0.96716	885.31	12848	
28	2.17403	100.00000	869.14	12901	+++++
29	2.13129	1.07525	852.05	12957	
30	2.01220	0.43865	804.44	13113	
31	1.59388	1.05014	637.21	13661	
32	0.04046	0.80271	16.17	15696	
33	0.00840	2.99922	3.36	15738	+
34	0.00000	66.66846	0.00	15749	+++++
35	-0.00840	2.66802	-3.36	15760	+
36	-0.04198	0.48176	-16.78	15804	

14-DEC-87 11:30:55

OSNUC 1H
 OBFRO 399.65 MHz
 OBSET 120.00 kHz
 OBFIN 14811.2 Hz
 PW1 3.0 us
 PW2 33.0 us
 PHS

FRACTION 3 FROM HPLC OF AGAR/10, PETROL ETHER/DIETHYL ETHER 95:5 VOL.

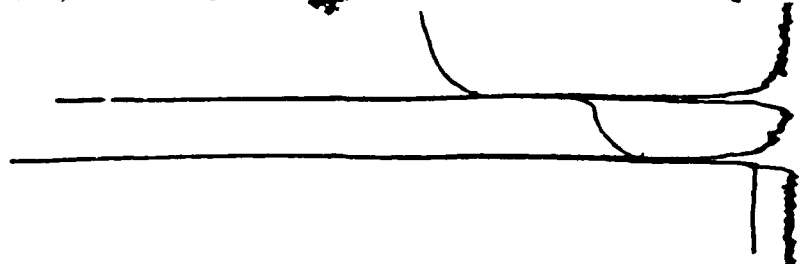
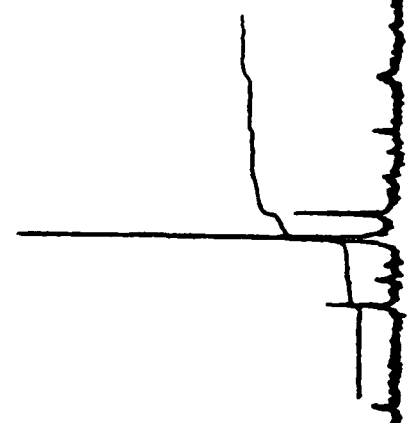
OBS IN 400 MHz.

SPECTRUM 3

$C_{10}H_4Cl_{12}$ 46% } MASS SPEC.
 $C_{10}H_3Cl_{13}$ 54% } ESTIMATE

R_2CHCL

R_2CH_2



14-DEC-87 12:10:41

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 MXINT 666973700
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 COMNT #5286/GREGORY/3/CDCL3/1H/C5/+25DEG/SGNON/SCH

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1	7.26103	28.23798	2902.83	6230	++++++ CHCl_3
2	6.03813	1.36029	2413.94	7832	
3	5.94806	7.04529	2377.93	7950	+ } R_2CHCl
4	5.91523	1.98020	2364.81	7993	
5	3.66410	14.72423	1464.84	10942	+++ } R_2CH_2
6	3.58090	13.83143	1431.58	11051	+++ }
7	2.70457	2.49464	1081.24	12199	
8	2.17327	0.90036	868.84	12895	-
9	1.54885	3.20507	619.20	13713	+
10	1.53969	1.82560	615.54	13725	
11	1.52213	1.07773	608.52	13748	
12	1.50305	3.15426	600.89	13773	+
13	1.32900	0.85171	531.31	14001	
14	1.30763	1.28600	522.77	14029	
15	1.28931	1.61713	515.44	14053	
16	1.27099	1.83422	508.12	14077	
17	1.25266	1.81832	500.79	14101	
18	1.22137	0.63397	488.28	14142	
19	1.17022	1.03425	467.83	14209	
20	1.15343	1.58423	461.12	14231	
21	1.13129	1.40567	452.27	14260	
22	1.10457	0.71636	441.59	14295	
23	0.89389	3.47061	357.36	14571	+
24	0.88320	2.23065	353.09	14585	
25	0.87557	8.55405	350.04	14595	++ }
26	0.87099	13.49026	348.21	14601	+++ } PETROL ETHER
27	0.85496	15.06149	341.80	14622	+++ }
28	0.85038	5.16451	339.97	14628	+
29	0.84427	4.46447	337.52	14636	+
30	0.83816	3.86436	335.08	14644	+
31	0.83358	4.44974	333.25	14650	+
32	0.82748	3.87931	330.81	14658	+
33	0.06870	1.10554	27.47	15652	
34	0.03435	1.06598	13.73	15697	
35	0.02214	0.74910	8.85	15713	
36	0.00687	5.44087	2.75	15733	+
37	0.00000	100.00000	0.00	15742	+++++
38	-0.00840	4.22455	-3.36	15753	+
39	-0.01985	1.91707	-7.93	15768	
40	-0.03511	1.43835	-14.04	15788	

14-DEC-87 12:13:12

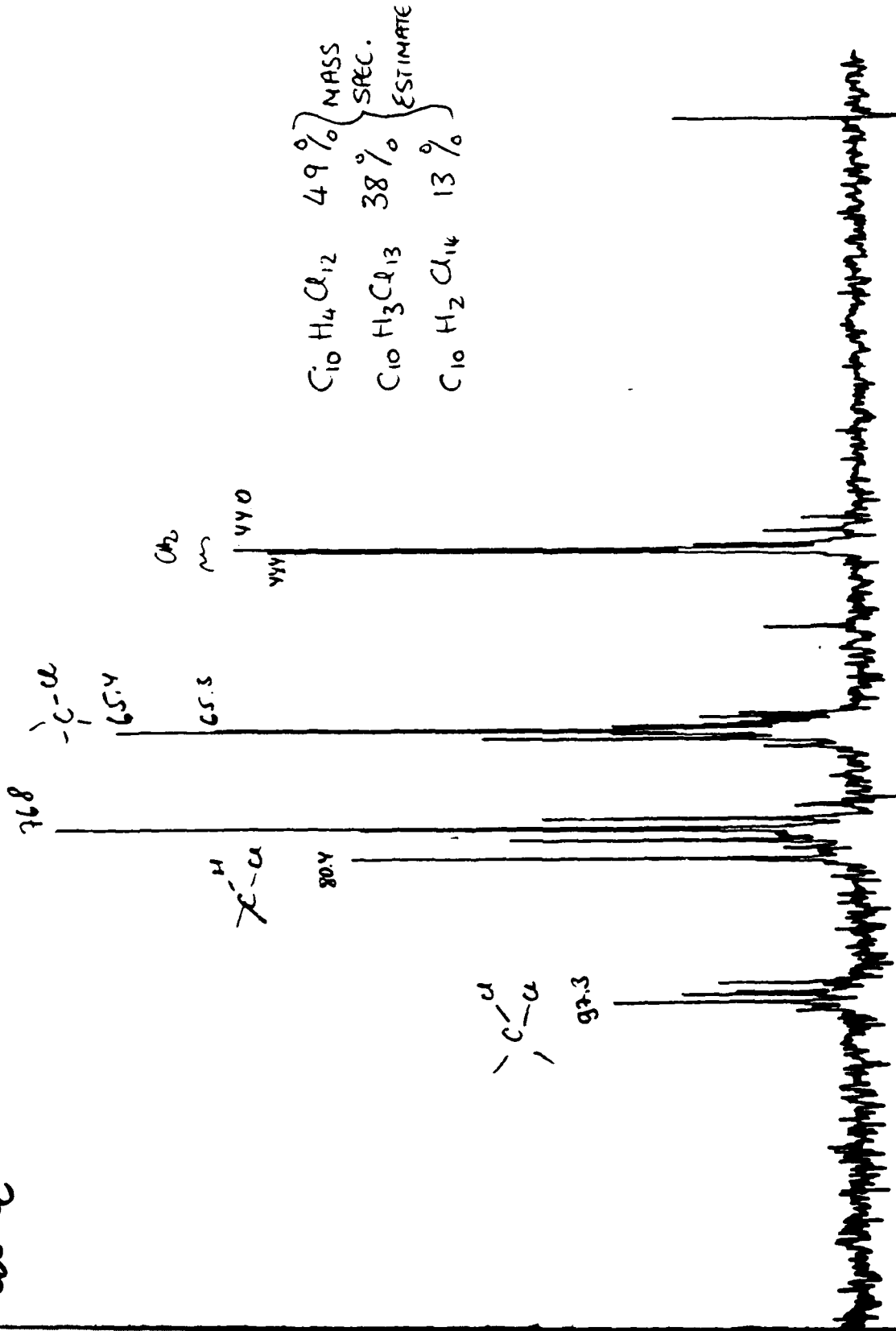
OBNUC 1H
 OBFRO 399.65 MHz
 OBSET 120.00 kHz
 OBFIN 14811.2 Hz
 PW1 3.0 us
 PW2 33.0 us

POLYCHLOROADAMANTANE

SPECTRUM 4

PRE - HPLC

OBS ¹³C



NUCLEUS ¹³C

OBS. _____

LOCK _____

IRR. _____

FREQUENCY _____

OBS. _____

LOCK _____

IRR. _____

PULSE ☐ SINGLE ☐ MULTI

WIDTH _____ μ SEC. ()

INTERVAL _____ SEC.

REPETITION _____ SEC.

DATA POINTS _____

WINDOW _____

REQ. RANGE _____ M

FILTER B.W. _____ M

AMPLITUDE R.F. _____ A.F.

NO. OF SCANS _____

DECOUPLING ☐ C.W. _____

☐ NOISE _____

☐ EXT. _____

POWER _____

LOCK LEVEL R.F. _____ A.F.

DATE _____

OPERATOR _____

REMARKS _____

ZERO 1.3105 SEC : 6250.00 HZ CONST
 C1 = 200 SCANS :
 C0.7 QUIK:5 TYPE 1:
 R-RATE SIGNAL FILE IS FROM NO. 66 TO NO. 70 MOVE:70 DISP:H MOVE:66 DISP
 H-DISP:H\$STOR+REDUC
 FREQ.RANGE= 6250.00 HZ 248.508PPM
 RESOLUTION= 0.76 HZ
 TOTAL PEAK= 20 NOISE(%)= 2.89 REFERENCE (PPM)= 76.959 : 77

NO.	POSIT	FREQ (HZ)	PPM	HEIGHT (%)
1	66	5821.99	231.481	-18.000
2	4491	2445.98	97.253	16.370
3	4520	2423.85	96.374	11.902
4	4530	2416.22	96.068	8.148
5	4566	2388.76	94.976	9.515
6	5047	2021.78	80.386	33.223
7	5117	1968.38	78.265	23.097
8	5159	1936.34	76.991	32.692
9	5164	1932.52	76.838	52.206
10	5201	1904.29	75.717	20.893
11	5509	1669.31	66.374	24.751
12	5530	1653.28	65.735	16.224
13	5540	1645.66	65.432	48.177
14	5544	1642.60	65.310	41.610
15	5559	1631.16	64.857	16.120
16	5592	1605.98	63.854	10.486
17	6235	1115.41	44.350	38.289
18	6246	1107.02	44.015	40.474
19	6262	1094.81	43.530	10.687
20	7932	-179.29	-7.129	11.933

768

804

$R_2 \text{ CCL}_2$

$R_3 \text{ CCL (BRIDGEHEADS)}$

$R_2 \text{ CH}_2$

DISP:V-? MOVE:66 CONST

C1 = 200 SCANS : 2000
 C ACCUM CONTINUE

92.3

DATE
FILMED
8 8