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Figure 4. ^{29}Si INEPT spectrum of poly(phenylsilane)

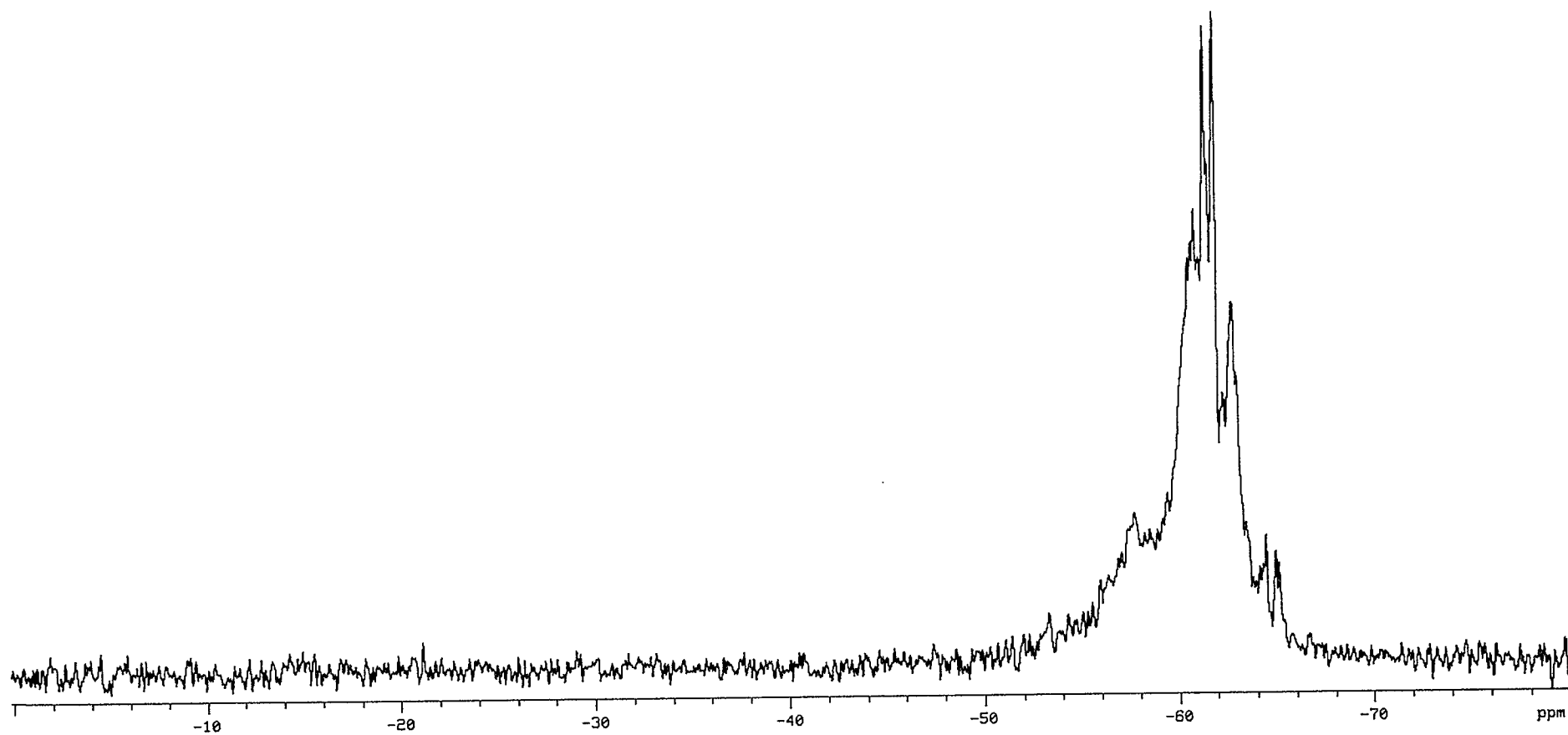


Figure 5. ^{29}Si INEPT spectrum of oxidized material

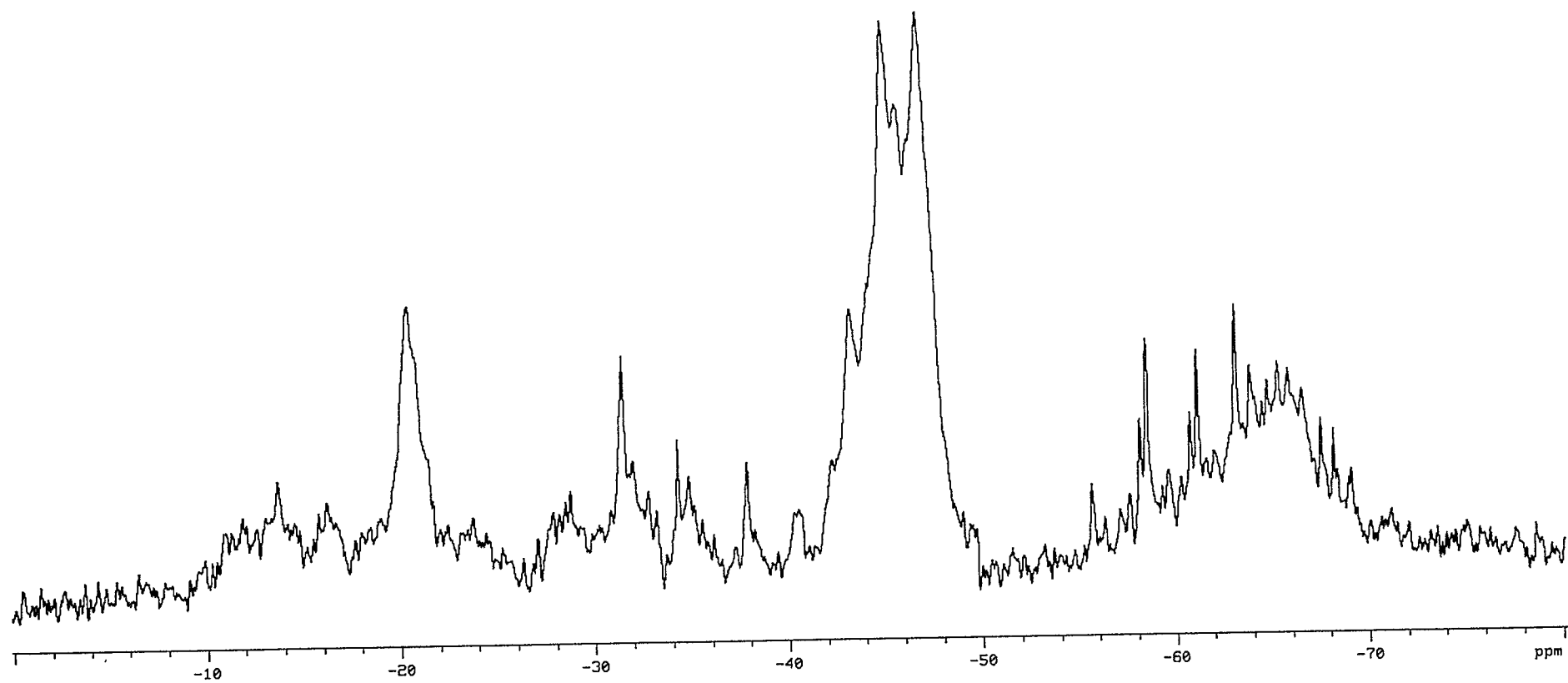


Figure 6. IR spectrum of poly(phenylsilane)

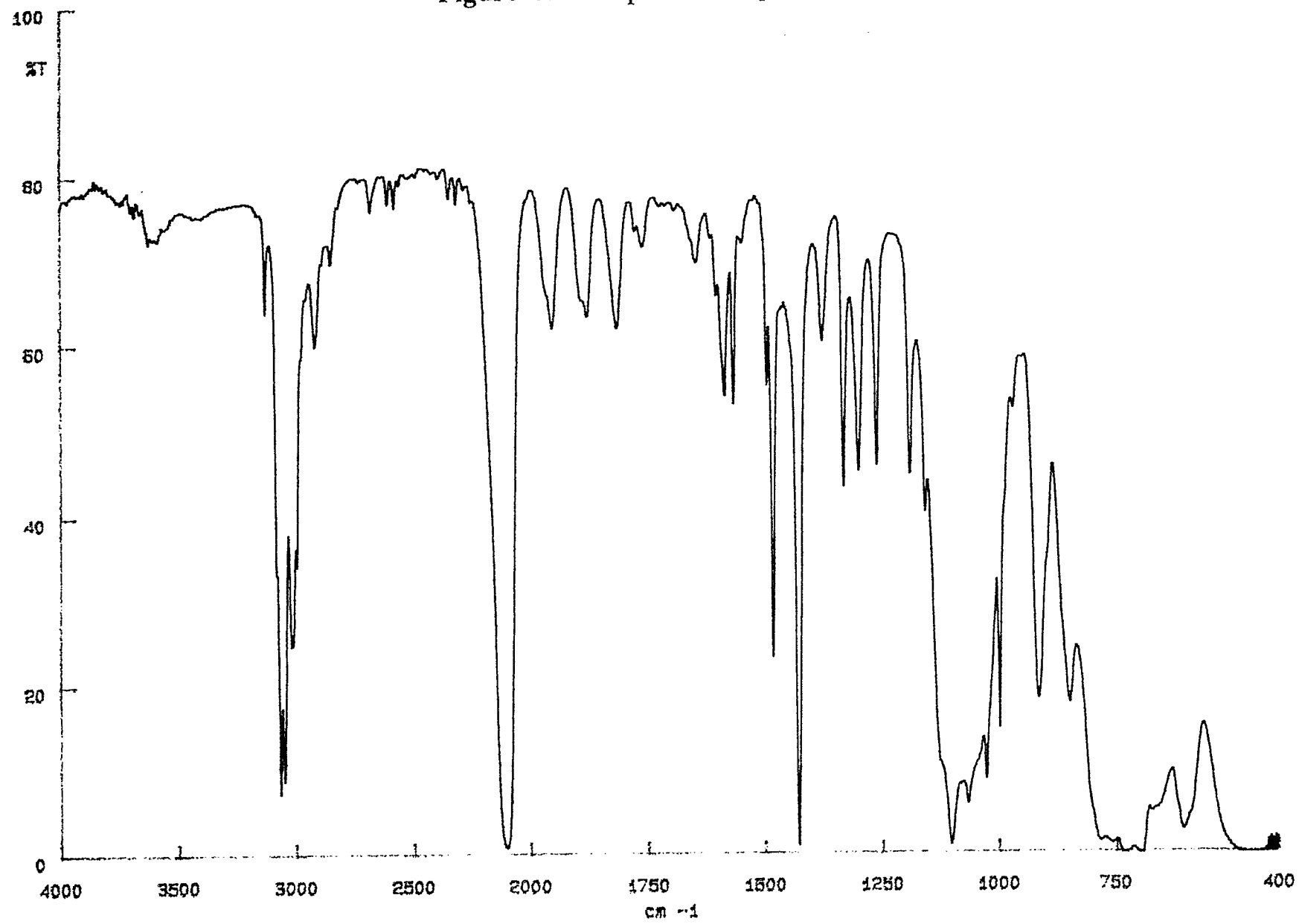
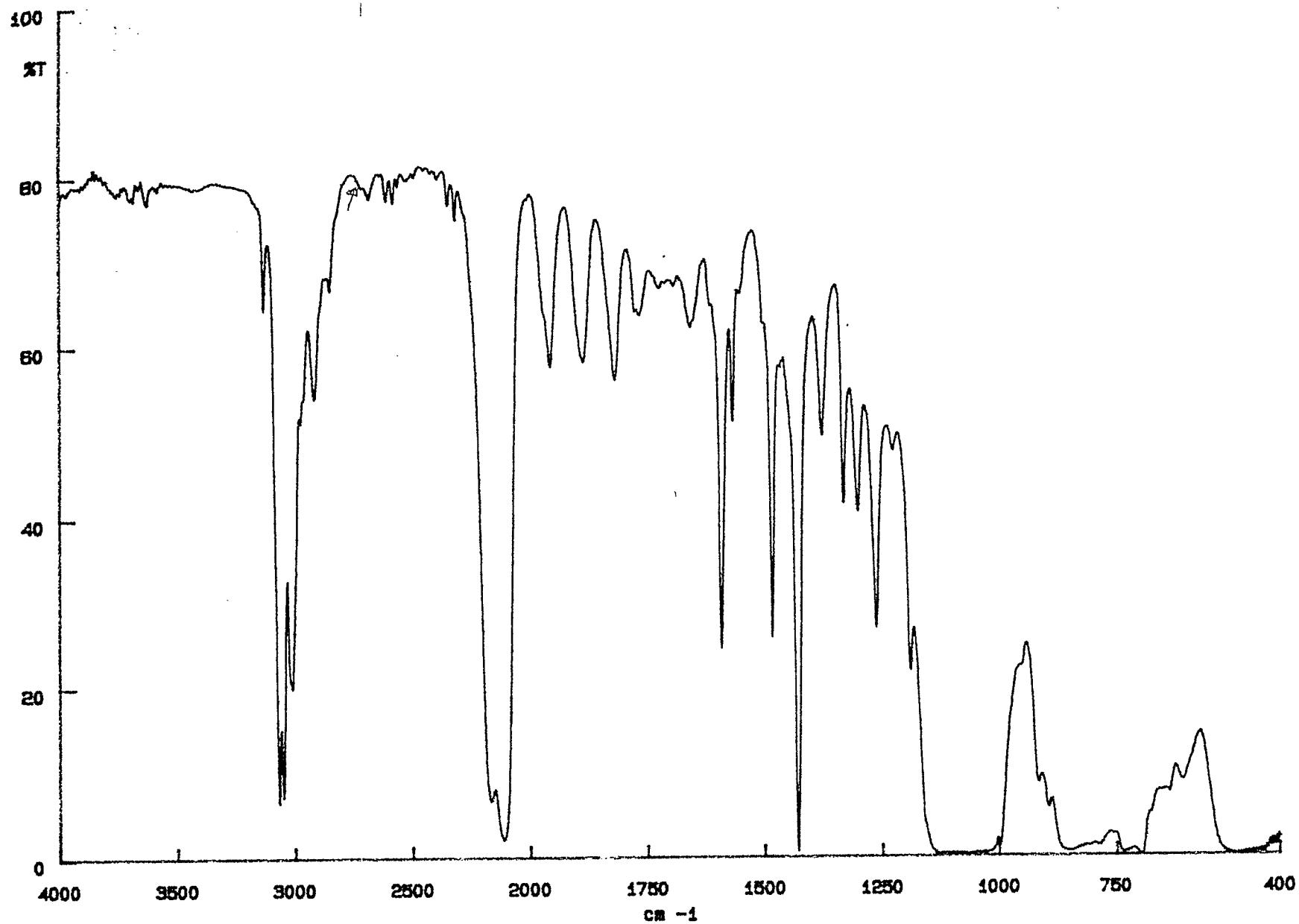


Figure 7. IR spectrum of oxidized material



Reaction of $(\text{Me}_3\text{Si})_2\text{Si}(\text{H})\text{Me}$ with oxygen-labelling mixture

(55.4% ^{16}O - ^{16}O / 0.8% ^{16}O - ^{18}O / 43.8% ^{16}O - ^{18}O)

Calculation of label distribution in the products from mass spectroscopic data

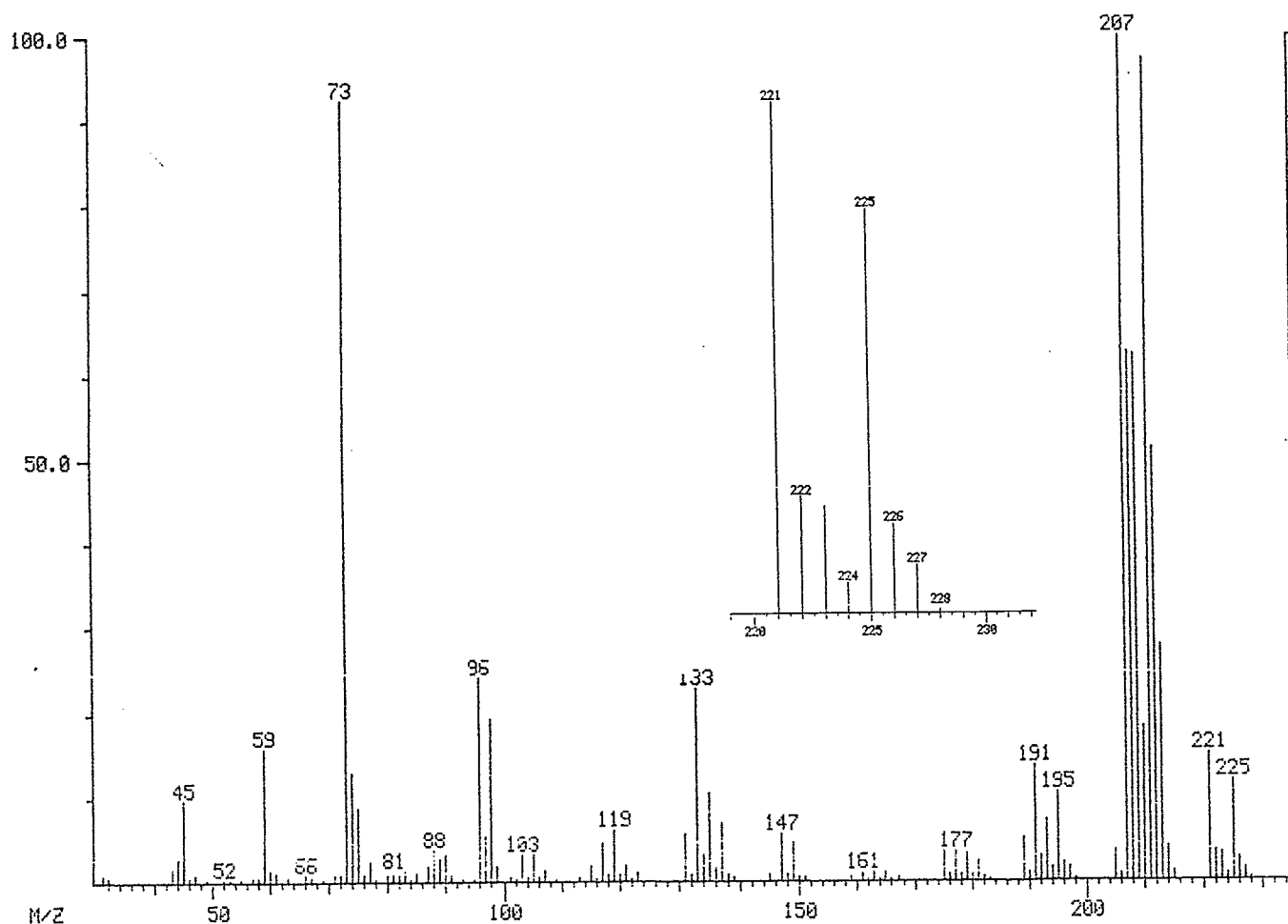


Figure 8. Electron impact mass spectrum of coeluting isotopomers of $(\text{Me}_3\text{SiO})_2\text{Si}(\text{H})\text{Me}$ ($\text{C}_7\text{H}_{22}\text{O}_2\text{Si}_3$, monoisotopic nominal mass 222 Da) obtained by GC/MS analysis of the reaction mixture. The insert shows the isotopic cluster of $[\text{M}-1]^+$.

Table 3. Relative abundances^a within the isotopic cluster of [M-1]⁺ in the EI mass spectra of coeluting isotopomers of (Me₃SiO)₂Si(H)Me, recorded in successive scans of the GC/MS analysis of the reaction mixture.

Scan Number	m/z					
	221 ^b	222 ^c	223 ^d	225 ^e	226 ^f	227 ^g
212	100	21.4	19.5	80.7	18.6	9.7
213	100	22.4	21.9	82.8	18.6	9.4
214	100	23.8	21.8	83.7	18.9	10.3
215	100	22.6	21.5	76.2	16.0	9.8
216	100	22.5	20.1	76.6	17.1	9.1
Aver.	100	22.5	21.0	80.0	17.8	9.7
St. Dev.	-	0.85	1.1	3.4	1.2	0.45

^aNormalized with respect to m/z 221. ^bC₇H₂₁O₂Si₃. ^cC₇H₂₁O₂Si₂²⁹Si and C₆¹³CH₂₁O₂Si₃. ^dC₇H₂₁O₂Si₂³⁰Si and C₇H₂₁O¹⁸OSi₃. ^eC₇H₂₁¹⁸O₂Si₃. ^fC₇H₂₁¹⁸O₂Si₂²⁹Si and C₆¹³CH₂₁¹⁸O₂Si₃. ^gC₇H₂₁¹⁸O₂Si₂³⁰Si.

The contribution to the relative abundance of m/z 223 due to the ion C₇H₂₁O₂Si₂³⁰Si can be calculated as follows:

$$A\%(C_7H_{21}O_2Si_2^{30}Si)/A\%(C_7H_{21}O_2Si_3) = A\%(C_7H_{21}^{18}O_2Si_2^{30}Si)/A\%(C_7H_{21}^{18}O_2Si_3)$$

rearranging and substituting the values reported in Table II:

$$A\%(C_7H_{21}O_2Si_2^{30}Si) = 12.1 \pm 0.5 \%$$

When this value is subtracted from the experimental one (A%(m/z 223)_{EXP} = 21.0 ± 1.1 %), the contribution to A%(m/z 223)_{EXP} due to the ion [C₇H₂₂O¹⁸OSi₃]⁺ can be obtained:

$$A\%(C_7H_{21}O^{18}OSi_3) = 9.1 \pm 1.2 \%$$

Since the major source of this ion is loss of H· from the molecular ion [(Me₃SiO)(Me₃Si¹⁸O)Si(H)Me]⁺, the corresponding neutral molecule is present in the reaction mixture. The ratio between the three isotopomeric products in the reaction mixture is:

$$(Me_3SiO)_2Si(H)Me : (Me_3SiO)(Me_3Si^{18}O)Si(H)Me : (Me_3Si^{18}O)_2Si(H)Me = \\ = 100 : 9.1 : 80$$

Therefore, the amount of the species having one ¹⁶O and one ¹⁸O, relative to the total amount of the three isotopomers, can be calculated to be 4.8 %

Table 4. Oxygen concentration during the AIBN (0.026M) initiated autoxidation at 50 °C of (Me₃Si)₂Si(H)Me (0.3M) in benzene in the presence of α -tocopherol (2×10^{-4} M) (cf. Figure 2a)

Time / s	[O ₂] / M	Time / s	[O ₂] / M
0	0.00190	3006	0.00148
167	0.00187	3173	0.00147
334	0.00185	3340	0.00145
501	0.00182	3507	0.00144
668	0.00182	3674	0.00143
835	0.00177	3841	0.00142
1002	0.00176	4008	0.00140
1169	0.00172	4175	0.00138
1336	0.00175	4342	0.00135
1503	0.00172	4509	0.00132
1670	0.00170	4676	0.00130
1837	0.00167	4843	0.00128
2004	0.00165	5010	0.00126
2171	0.001625	5177	0.00102
2338	0.00160	5344	0.00086
2505	0.00157	5511	0.00056
2672	0.00155	5678	0.00034
2839	0.00150	5845	0.00007 ₅
		6012	0

**Table 5. Oxygen concentration during the AIBN (0.026M)
initiated autoxidation at 50 °C of (Me₃Si)₂Si(H)Me (0.3M) in
benzene (cf. Figure 2a)**

Time/s	[O ₂] / M	Time/s	[O ₂] / M
0	0.00190	522	0.00102
43.5	0.00182	565.5	0.00093
87	0.00174	609	0.00077
130.5	0.00171	652.5	0.00070
174	0.00157	696	0.00061
217.5	0.00150	739.5	0.00043
261	0.00147	850	0.00032
304.5	0.00142	950	0.00025
348	0.00134	1000	0.00016
391.5	0.00125	1100	0.000025
435	0.00113		
478.5	0.00107		

Table 6. Oxygen concentration during the di-*tert*-butyl peroxide (0.017M) initiated autoxidation at 50 °C of (Me₃Si)₂Si(H)Me (0.26M) in benzene in the presence of α -tocopherol (1.4×10^{-4} M) (cf. Figure 2b)

Time/s	[O ₂] / M	Time/s	[O ₂] / M
0	0.00190	971	0.00136
98	0.00187	1068	0.00130
195	0.00184	1165	0.00120
292	0.00180	1262	0.00110
389	0.00173	1359	0.00090
486	0.00169	1456	0.00070
583	0.00164	1553	0.00046
680	0.00157	1650	0.00016
777	0.00152	1720	0.00001 ₂
874	0.00146		

Table 7. Oxygen concentration during the di-*tert*-butyl peroxide (0.017M) initiated autoxidation at 50 °C of (Me₃Si)₂Si(H)Me (0.26M) in benzene (cf. Figure 2b)

Time/s	[O ₂] / M
0	0.00190
88	0.00176
175	0.00138
263	0.00056
350	0.00015
438	0