



NUCLEAR MAGNETIC RESONANCE
SPECTRA OF TRIALKYL PHOSPHATES
AND RELATED COMPOUNDS
I. Proton Magnetic Resonance Spectra

by

W. E. Shuler and R. C. Axtmann

Separations Chemistry Division

June 1960

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Printed in USA. Price \$1.50
Available from the Office of Technical Services
U. S. Department of Commerce
Washington 25, D. C.

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DP - 474

CHEMISTRY - GENERAL
(TID-4500, 15th Ed.)

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I. PROTON MAGNETIC RESONANCE SPECTRA

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Woodfin E. Shuler and Robert C. Axtmann*

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E. I. du Pont de Nemours & Co.
Explosives Department - Atomic Energy Division
Technical Division - Savannah River Laboratory

Printed for
The United States Atomic Energy Commission
Contract AT(07-2)-1

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ABSTRACT

High resolution proton magnetic resonance spectra are presented for 48 trialkyl phosphates and related phosphorus compounds. Chemical shifts, spin-spin coupling constants, and spectral analyses are reported for representative compounds of interest in solvent extraction technology.

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NUCLEAR MAGNETIC RESONANCE SPECTRA OF TRIALKYL PHOSPHATES AND RELATED COMPOUNDS

I. PROTON MAGNETIC RESONANCE SPECTRA

INTRODUCTION

Tributyl phosphate (TBP) is the solvent most commonly used in solvent extraction processes for the purification and recovery of uranium and plutonium from irradiated fuels. Although the TBP solvent extraction processes have been well characterized from the standpoints of stoichiometry, mass transfer, and stability of the solvent, the molecular structure of the complexes involved in the processes has received relatively little attention. Comyns pointed out in a recent review⁽¹⁾ that no determination has been made of the structure of a complex as common and important as the one formed by the addition of uranyl nitrate to TBP. In spite of the fact that nitric acid is present in nearly all TBP processes, the TBP·H₂O·HNO₃ system has never been properly characterized and several recent authors^(2,3) have disagreed as to whether TBP associates with one, two, or more molecules of nitric acid.

The present paper reports a portion of the effort to characterize TBP and its complexes by means of optical and nuclear magnetic resonance (NMR) spectroscopy. An NMR study of the TBP-water system has already been reported⁽⁴⁾. Axtmann, Shuler, and Eberly studied the reflection of the inductive effect on the spin-spin coupling interaction between P³¹ and the α protons in TBP and other trialkyl phosphates⁽⁵⁾.

Other work in progress bears on the relationship of the thermodynamics of extraction of uranyl nitrate to chemical shifts and coupling constants, and the behavior of the chemical shift of the protons undergoing exchange in solutions of H₂O, D₂O, and HNO₃ in TBP.

SUMMARY

Proton NMR spectra were obtained for TBP and 27 other trialkyl phosphates, several trialkyl phosphites, phosphines, phosphorothioates, phosphonates, and tri-n-butylphosphine oxide. Spectra representing the different classes of compounds were analyzed to first order, and chemical shifts, δ , and spin-spin coupling constants, J, were determined.

DISCUSSION

EXPERIMENTAL

All of the NMR spectra were obtained at room temperature with samples rotated in coaxial sample tubes*. The spectrometer was a Varian Model 4300B that was equipped with a "superstabilized" 12-inch magnet

* Wilmad Glass Co., Landisville, New Jersey

system. Chemical shifts were measured by the sideband technique to ± 0.5 cps relative to benzene as an external reference in the outer sample tube. About 5 volume per cent benzene was added to the compound in the inner tube after the high resolution spectra were recorded, and new spectra were run to obtain the "solvent shift" or separation between the internal and external benzene reference signals. The spectrometer's Sanborn recorder was used together with sidebands for some of the shift measurements, while others were measured by sideband calibration of spectra recorded on a Varian G-10 recorder.

The spin-spin coupling constants of highest precision in Table I were determined from G-10 recordings for which the known splitting of the methylene group (4.8 cps)⁽⁶⁾ of ethyl alcohol was used to calibrate the magnetic sweep speed before and after each measurement. The reproducibility of these measurements on successive traces was ± 0.2 cps. The spin-spin coupling constant for trimethyl phosphate was determined by the "wobble-beat" method.^(7,8) The remaining coupling constants in Table I were determined by measurement of slow recordings (~ 0.2 cps/second) that were calibrated by the sideband technique.

Some of the phosphorus compounds were obtained commercially and used without further purification. T. H. Siddall, III, of this laboratory, synthesized the other compounds whose spectra are reported.⁽⁹⁾

RESULTS

The high resolution spectra and, for some compounds, the theoretical spectra based on first-order coupling of spins are given in the Appendix. The protons on the carbon atom adjacent to the ester oxygen -- or, if no oxygen atom is interposed between the alkyl chain and the phosphorus atom, the protons on the carbon bonded to the phosphorus atom -- are designated as α protons. Those protons on the second carbon from the phosphorus atom are β protons, etc. Signals known to result from water contained in the sample and spurious signals are shaded in the spectra. Reference signals from internal and external references and sidebands are designated by R_I , R_E , and R_S , respectively.

The chemical shifts given in Table I are displacements from an external benzene reference and have not been adjusted for bulk magnetic susceptibility.

The spectra of several of the smaller molecules yield a complete set of proton coupling constants. For example, $J_{\alpha H-\beta H}$, $J_{\alpha H-P}$, and $J_{\beta H-P}$ were obtained for triethyl phosphate. The sharp decrease in J_{H-P} in going from the α protons to the β protons (from 8.38 cps to 0.76 cps for triethyl phosphate) constitutes experimental evidence for the rapid attenuation of electron spin coupling of nuclei as the number of intervening bonds increases.

The decrease in $J_{\alpha\text{H}-\text{P}}$ with increasing alkyl chain length of several tri-n-alkyl phosphates has been correlated with the inductive effect⁽⁵⁾

The data indicate that in carbon chains containing about six or seven atoms the coupling of nuclei through the spin of their electrons is very slight; extrapolation was made of the $J_{\text{H}-\text{H}}$ values for adjacent methylene groups estimated from Spectrum 10 for α , β , γ , and δ protons in tri-n-amyI phosphate. The conclusion that these methylene groups are devoid of any intrinsic difference in chemical properties is demonstrated in a less precise manner by the fact that the protons in methylene groups that are several carbons removed from the electro-negative substituent show indistinguishable chemical shifts, i.e., magnetic equivalence.

ANALYSIS OF THE HIGH RESOLUTION PROTON SPECTRA

The fields at which resonance occurs are slightly different for the several chemically distinguishable groups of protons in a given molecule. Thus, the protons of a methyl group resonate at a higher field than do those of a neighboring methylene group if both groups are irradiated at the same fixed frequency. These small differences, termed "chemical shifts", are proportional to the applied field strength. In this paper the shifts are reported in cycles per second above the proton resonance of an external benzene reference at 40 megacycles per second. Shielding numbers are shown on the spectra. The shielding number is independent of applied field strength and equal to $(\text{H}-\text{H}_\text{r})/\text{H}_\text{r}$, where H is the resonance field of the sample signal and H_r is the resonance field of the reference benzene signal with both H and H_r being determined at the same frequency.

The resonances of the magnetically equivalent protons in a chemical group (e.g., methylene group of ethyl alcohol) often exhibit a field-independent fine structure if there are nuclei with nonzero spin within the distance of three or four chemical bonds. The fine structure arises from spin-spin interactions via the valence electrons of the intervening atoms. The relations governing the number and intensity ratios of the fine structure of multiplet signals are quite simple in the cases of molecules that contain only spin 1/2 or spin zero nuclei⁽¹⁰⁾. If N is the number of chemically (and magnetically) equivalent spin 1/2 neighbors of a given chemical group, to first order the number of multiplet signals will be $(N + 1)$, and the signal intensities will be in the ratios of the coefficients of the binomial expansion of $(a + b)^N$. If, in addition, there is a second group of M near neighbors, each of the $(N + 1)$ signals will be split into $(M + 1)$ subsignals and the intensity ratios of the new subsignals will be as the coefficients of the binomial expansion of $(a + b)^M$.

When, by a combination of instrumental and molecular factors, "super" high resolution occurs, additional signals appear as splittings of those that appeared at lower resolution. The simple rules of first-

order multiplet analysis given in the preceding paragraph then break down, and more sophisticated analyses such as those applied to a number of simple organic compounds by Arnold⁽⁶⁾ and Anderson⁽¹¹⁾ are required. The fine structure of a number of the compounds studied here may be analyzed with the first-order rules.

One of the purposes of the present work was to analyze the proton NMR spectrum of tributyl phosphate. As in most problems of spectroscopic assignment it was necessary to examine the spectra of a number of closely related compounds. Phosphorus compounds were selected on the basis of the length of the alkyl chain and the class of compound. By selection of compound class, the atom adjacent to the phosphorus atom was varied from no atom (phosphines and phosphites) to oxygen (phosphates and phosphonates) and sulfur (phosphorothioates). Since several of these compounds have shown promise as alternate extractants for various radiochemical separation processes⁽⁹⁾, the spectra of all as well as representative analyses are presented here. The spectrograms all show the external benzene reference as a single signal at the low field end.

1. Trimethyl Phosphate, (CH₃O)₃PO (Spectrum 1)

The protons on the methyl groups give a symmetrical doublet that arises from spin-spin interaction with the single phosphorus nucleus. No evidence is seen for rotational isomerism, which would be manifested in this spectrum as a further splitting of the two signals of the doublet. Mortimer has explained the doublet observed in the fundamental stretching band at 1290 cm⁻¹ for the phosphoryl bond of trimethyl phosphate by invoking rotational isomerism⁽¹²⁾. Although the present results do not confirm Mortimer's suggestion, they do not rule out the possibility of rotational isomerism, for, as Pople has pointed out recently⁽¹³⁾, there is no guarantee that the protons of any given pair of rotational isomers will be magnetically nonequivalent or that the rotational isomers will have lifetimes long enough that they may be observed by NMR.

2. Triethyl Phosphate, (C₂H₅O)₃PO (Spectrum 2)

The signal from the methyl protons appears as a triplet at the high field end of the spectrum. The splitting of the central signal of the triplet could be due to rotational isomerism but is much more likely to result from an extremely weak spin-spin interaction with P³¹, which is four chemical bonds removed compared with three in the case of trimethyl phosphate. A temperature study would be required to confirm the point. The apparent quintet at the low field end of the spectrum is the signal from the methylene protons. Close inspection of the spectrum demonstrates that the three central signals of the quintet are split approximately equally, making a total of eight multiplet signals. This fine structure can be accounted for by assuming that the methyl protons interact with the methylene protons

to produce four multiplet signals in the ratios 1:3:3:1; and that the single P^{31} nucleus then splits each of these signals into a doublet. The "theoretical" spectra on this basis would be as shown in Spectrum 2B, which agrees quite well with the observed spectrum.

3. Triethyl Phosphite, $(C_2H_5O)_3P$ (Spectrum 31)

Superficially at least, the spectrum of this compound is similar to that of triethyl phosphate. Spectrum 31B shows, however, that the methylene protons produce a multiplet with extensive fine structure; at least 16 signals are apparent whereas only 8 appeared with the phosphate. The simple first-order rules for intensity ratios and number of signals are not very useful at this stage of resolution. A second or perhaps even a third-order perturbation calculation such as that done for ethyl alcohol⁽⁶⁾ is required to account for all the signals that are observed.

The phosphite gives better resolution than the phosphate under the same experimental conditions because the phosphite has a significantly lower molecular dipole moment. The phosphate molecule, with its highly polar phosphoryl group, is held more rigidly within the liquid structure by induced dipolar attraction and is less free to tumble about. Free tumbling is necessary to average out the nuclear dipolar interactions to zero, which is necessary for the highest resolution⁽¹⁴⁾.

4. Triethyl Phosphorothioate, $(C_2H_5O)_3PS$ (Spectrum 29)

The spectrum for this compound is quite similar to that for the previous two compounds. Due to a favorable ratio of $J_{CH_2-P}/J_{CH_3-CH_2}$, the multiplet of the methylene protons appears as a clearly delineated octet (see discussion under triethyl phosphate). Finegold⁽¹⁵⁾ has noted that H^1-P^{31} spin-spin coupling is enhanced when the phosphorus atoms are attached to double-bonded sulfur atoms rather than to double-bonded oxygen atoms. This observation is borne out in the present case (8.38 versus 10.18 cps) as well as in tributyl phosphorothioate.

5. Tri-n-propyl Phosphate, $(C_3H_7O)_3PO$ (Spectrum 4)

The apparent quartet at the low field side of the spectrum is from the α methylene protons. Actually the apparent quartet is an unresolved sextet. The methyl protons give a triplet that is, characteristically, at the extreme high field end of the spectrum. It is noteworthy that this triplet does not have the 1:2:1 intensity ratio that would be predicted by the binomial coefficients rule. The high field branch of the triplet is quite low in intensity. In butyl compounds this branch disappears almost entirely (see Spectrum 6 and compare with Spectrum 2, the ethyl compound). The signal of the β methylene protons appears as a multiplet at slightly lower field than the methyl protons. This multiplet should have at least 12 signals; these are not well resolved in the spectrogram.

6. Tri-n-butylphosphine, $(C_4H_9)_3P$ (Spectrum 37)

Because no oxygen atoms are interposed between the phosphorus atom and the alkyl chains, there is very little chemical shift for the α methylene protons and the α , β , and γ methylene spectrum is grouped near the signal of the methyl protons. The protons from the methyl groups give an asymmetric doublet -- the high field branch of the expected triplet disappears in butyl compounds. The natural width of the methylene proton signal obscures the fine structure and no further analysis is possible.

7. Tri-n-butyl Phosphate $(C_4H_9O)_3PO$ (Spectrum 6)

The protons of the α methylene groups give a sextet -- the P^{31} interaction doubles the triplet from interaction with the pairs of nearest neighbor methylene protons. The small single signal located about 60 cps above the signal just described has been definitely identified as a water signal⁽⁴⁾. The intense signal at the extreme high field end of the spectra is at the usual methyl position. The remainder of the signals, from the β and γ methylene protons, (21 may be seen) are not well resolved. Attempts to improve the resolution by diluting the TBP in an inert solvent such as CCl_4 were unsuccessful.

8. Tri-n-butyl Phosphite, $(C_4H_9O)_3P$ (Spectrum 32)

The spectrum is very similar to that of TBP. The β and γ methylene signals are much better resolved, however, for the same reason that triethyl phosphite gave a higher resolution than did triethyl phosphate.

9. Tri-n-butyl Phosphorothioate, $(C_4H_9O)_3PS$ (Spectrum 30)

The sextet arising from the α methylene protons is much clearer in this compound than in TBP because the spin-spin coupling with the P^{31} nucleus is stronger (see discussion under triethyl phosphorothioate).

10. Di-n-butyl n-butylphosphonate $(C_4H_9O)_2(C_4H_9)PO$ (Spectrum 41)

This spectrum is very similar to that of TBP (Spectrum 6) except that the signal of the α methylene groups bonded to oxygen has relatively less intensity than does the corresponding α methylene signal of TBP. This is because there are only $2/3$ as many such protons in the present compound. The signal from the α methylene groups bonded directly to phosphorus is present in the partially resolved region at about 204 cps.

11. n-Butyl di-sec-butylphosphinate (C₄H₉O)(C₄H₉)₂PO (Spectrum 39)

Compared to TBP, the spectrum shows a diminution of intensity at 105 cps as noted above for di-n-butyl n-butylphosphonate. The attendant increase in intensity due to the increased number of alkyl groups bonded directly to phosphorus (no intervening oxygen atoms) appears at about 210 cps.

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APPENDIX

TABLE I

 CHEMICAL SHIFTS AND COUPLING CONSTANTS
 (Reproducibility ± 0.5 cps except as noted)

Spectrum No.	Compound	(a) δ_{ϕ}	δ_{OH}	$\delta_{\text{OH-F}}$	(b) $\delta_{\text{OH-}\beta\text{H}}$	(c) $\delta_{\text{OH-}\beta\text{H}}$	δ_{CH_3}	(d) δ_{solvent}
1	Phosphates (RO) ₃ PO	-	114.4	11.19 \pm 0.2	-	-	-	-28.4
2	Trimethyl phosphate	-	101.5	8.38 \pm 0.2	7.10	7.18	212.9	-24.4
3	Triethyl phosphate	-	-	-	-	-	-	-
4	Tris-(2,2,2-trichloroethyl) phosphate	-	-	-	6.35	-	227.4	-24.4
5	Tri-n-propyl phosphate	-	106.5	7.70 \pm 0.1	-	-	-	-
6	Triallyl phosphate	-	-	-	-	-	-	-
7	Tri-n-butyl phosphate	-	106.2	7.65 \pm 0.1	5.94	6.21	227.9	-24.1
8	Triisobutyl phosphate	-	114.2	6.60 \pm 0.1	6.60	6.07	224.5 \pm 2	-23.8
9	Tri-sec-butyl phosphate	-	90.1	6.17	6.17	6.36	230.6 \pm 2	-
10	Trimethyl phosphate	-	87.1	7.6?	-	-	227.8	-28.4
11	Tri-n-amy1 phosphate	-	105.1	7.62 \pm 0.2	5.76	5.74	191.5	-25.0
12	Tri-2-amy1 phosphate	-	85.7	-	-	-	228.4	-
13	Tri-3-amy1 phosphate	-	95.4 \pm 1.	7.30	5.50	6.21	228.5 \pm 2	-
14	Trineopentyl phosphate	-	119.8	5.24 \pm 0.1	-	-	227.5 \pm 2	-24.9
15	Tri-n-hexyl phosphate	-	105.7	7.32	5.70	-	228.2	-24.5
16	Tris-(4-methyl-2-amy1) phosphate	-	84.5	5.8?	5.8?	-	232.4	-25.0
17	Tri-4-heptyl phosphate	-	92.4	6.7 \pm 1	4.9 \pm 1	-	226.0	-
18	Tris-(2,4-dimethyl-3-amy1) phosphate	-	109.0 \pm 1	-	-	-	227.6	-
19	Tris-(1,1,7-trihydroperfluoroheptyl) phosphate	-	85 \pm 5	-	-	-	226.0 \pm 2	-
20	Tri-iso-octyl phosphate	-	102.5 \pm 1	-	5.3 \pm 1	-	234.9 \pm 2	-
21	Tris-(2-ethylhexyl) phosphate	-	105.5 \pm 1	-	-	-	226.7 \pm 2	-
22	Tricyclohexyl phosphate	-	66 \pm 1	-	-	-	190.0*	-30.3
23	Tri-c-tolyl phosphate	-4.2	84 \pm 1	-	-	-	196.5*	-27.2
24	Tri-m-tolyl phosphate	-7.5	-	-	-	-	188.2	-15.5
25	Tri-p-tolyl phosphate	-11.4	-	-	-	-	183.5	-18.7
26	Di-sec-butyl phenyl phosphate	-18.0	-	-	-	-	182.0	-26.2
27	Triphenyl phosphate	-21.3 \pm 1	85.0	-	-	-	233.9 \pm 2	-
28	Tribenzyl phosphate	-17.4	-	-	-	-	-	-
29	Phosphorothioates (RO) ₂ SPS	-28.0	64.5	8.30 \pm 0.1	-	-	-	-29.0
30	Triethyl phosphorothioate	-	99.5	10.18	7.11	7.16	210.9	-27.2
31	Tri-n-butyl phosphorothioate	-	103.0	8.86	6.12	-	225.7	-25.9

*Ring methylene groups

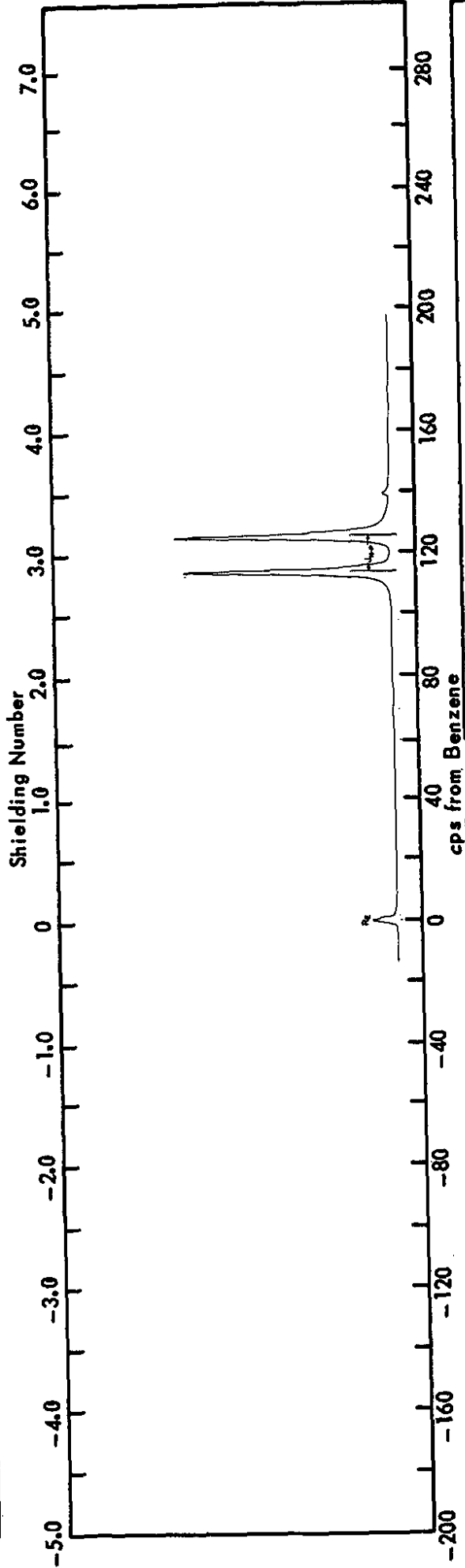
	<u>Phosphites (RO)₃P</u>							
31	Triethyl phosphite	-	119.0	7.90	6.87	6.90	223.2	-19.6
32	Tri-n-butyl phosphite	-	118.0	7.47	6.06	-	230.7	-18.4
33	Tridecyl phosphite	-	105 ±4	-	-	-	226.2	-
34	Triphenyl phosphite	0.00	-	-	-	-	-	-
35	Tri-p-tolyl phosphite	-6.02	-	-	-	-	190.8	-
36	Triethylene diphosphite	-	~100	-	-	-	-	-
	<u>Phosphines R₃P</u>							
37	Tri-n-butylphosphine	-	-	-	-	-	229.0	-
38	Triphenylphosphine	-27.6	-	-	-	-	-	-24.3
	<u>Phosphinates (RO)₂PO</u>							
39	n-Butyl di-sec-butylphosphinate	-	106.5	7.24	6.00	-	223.7	-28.4
40	sec-Butyl di-sec-butylphosphinate	-	86.6	6.17	6.17	-	225.0	-28.5
41	Di-n-butyl n-butylphosphonate	-	105.0	7.33	5.90	-	220.4	-23.7
42	Di-sec-butyl sec-butylphosphonate	-	91.8	-	-	-	227.8	-21.4
43	Di-n-amy l n-amy lphosphonate	-	104.9	-	-	-	227.8	-23.2
44	Bis-(2-ethylhexyl) chloromethylphosphonate	-	100.6 ±2	-	-	-	224.8	-24.4
45	Di-n-butyl cyclohexylphosphonate	-	101.5	7.39	-	-	224.5	-24.2
46	Di-n-hexyl n-hexylphosphonate	-	104.3	7.20	-	-	228.6	-23.4
47	Di-n-butyl n-tetradecylphosphonate	-	104.4	7.06	-	-	225.9	-24.7
	<u>Phosphine Oxides R₃PO</u>							
48	Tri-n-butylphosphine oxide	-	216.2	-	-	-	232.0	-16.4

- (a) Chemical shift of aromatic ring protons with reference to external benzene reference
(b) Coupling constant determined from a proton spectrum
(c) Coupling constant determined from β proton or CH₃ proton spectrum
(d) Chemical shift of internal benzene (~5% v) with reference to external benzene reference

Trimethyl phosphate

Proton Magnetic Resonance Spectral Data

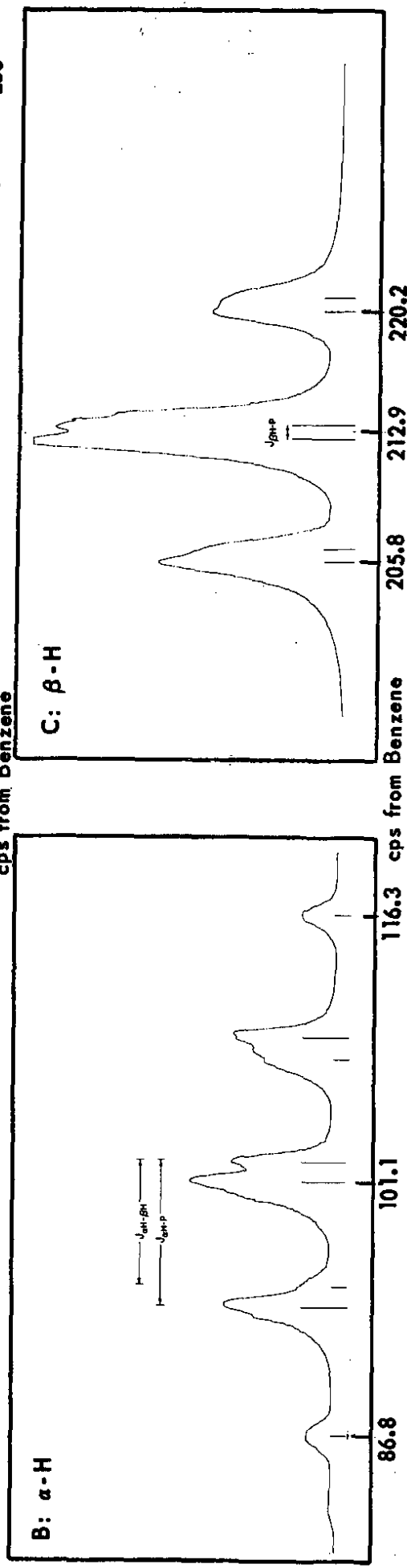
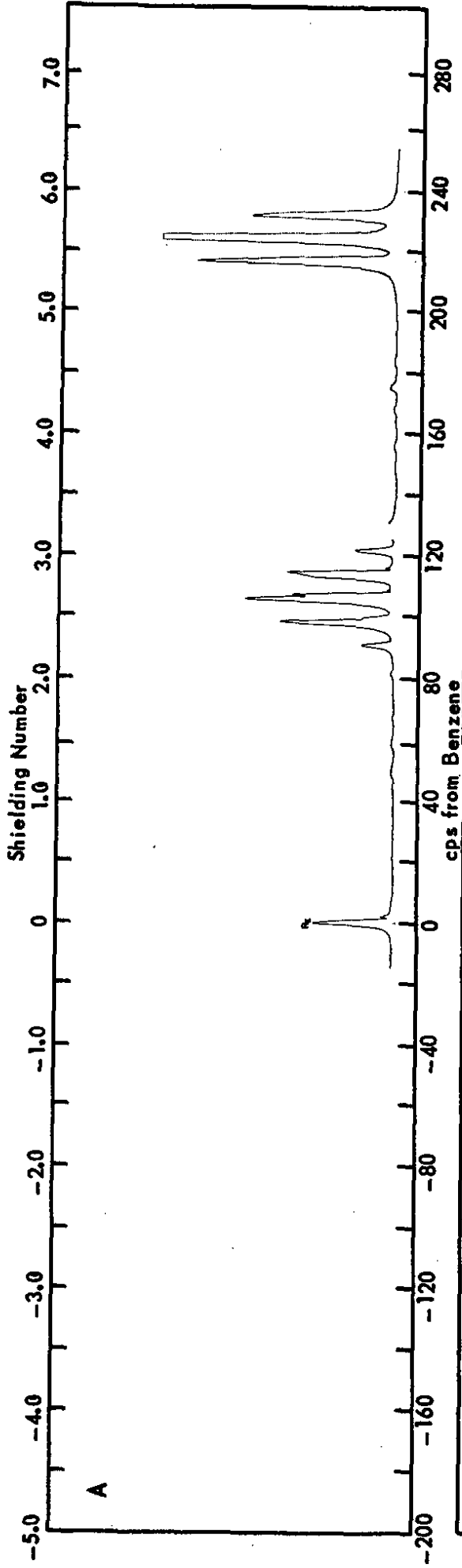
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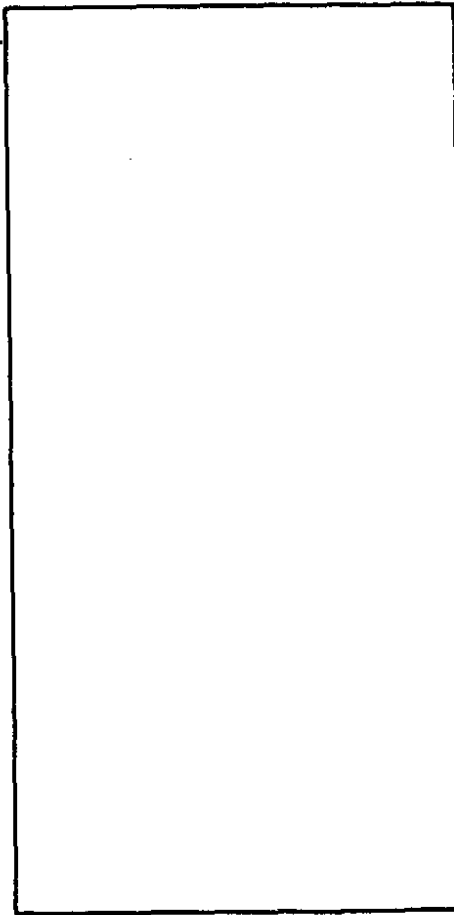
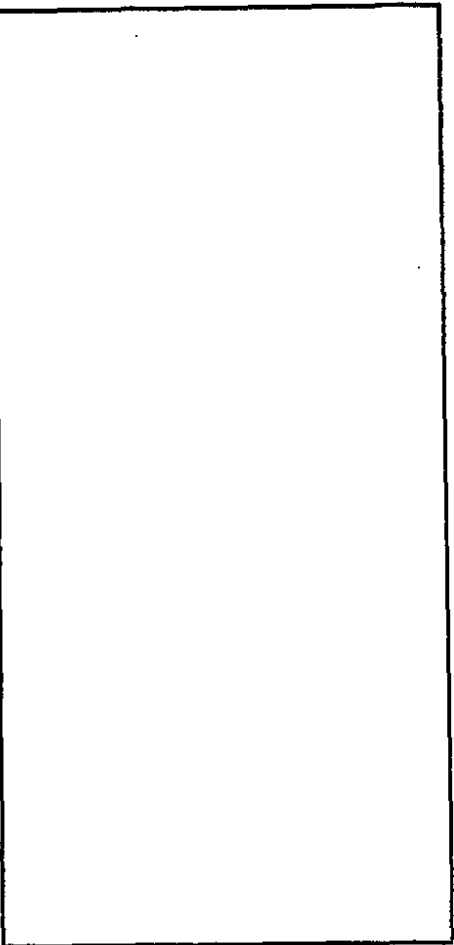
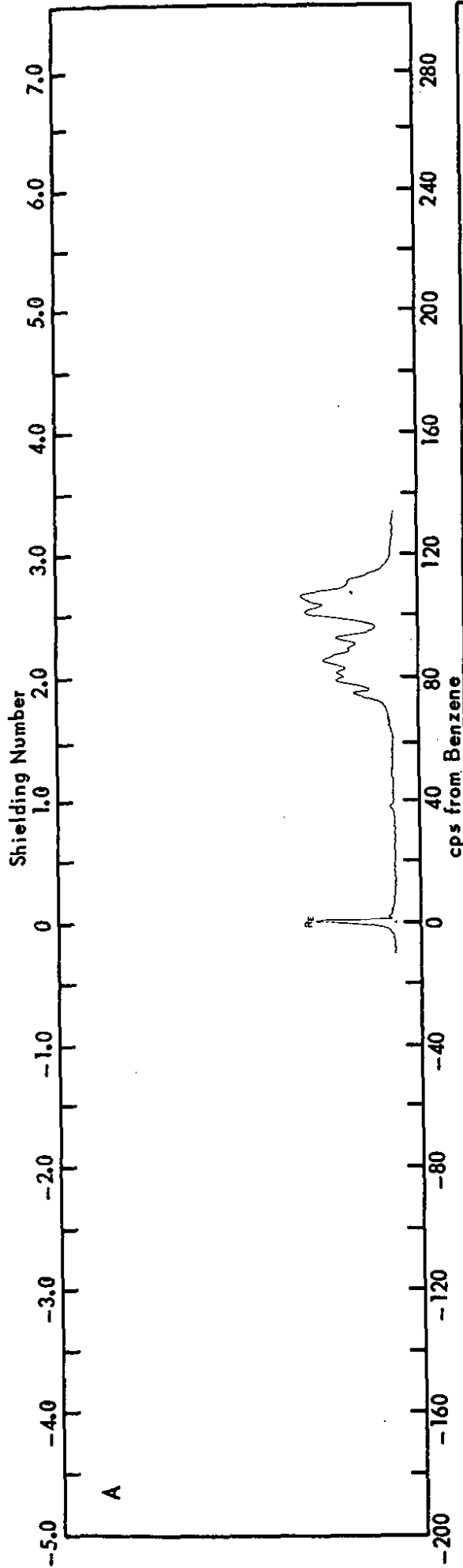
Resonance center 114.4 cps
Resonance center with internal benzene 118.0 cps

Resonance center 114.4 cps
Resonance center with internal benzene 118.0 cps

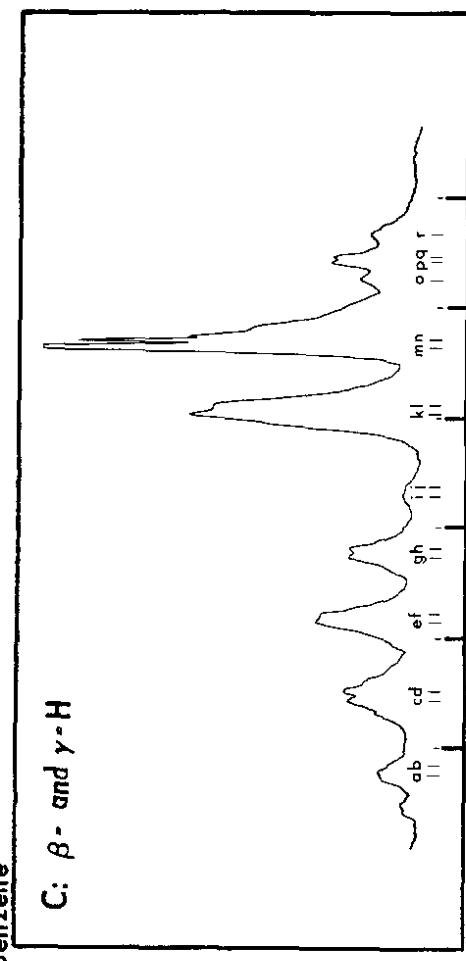
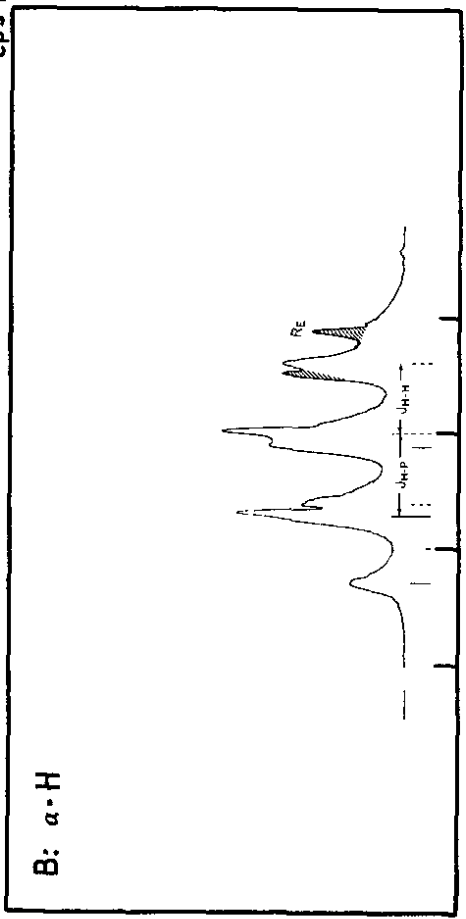
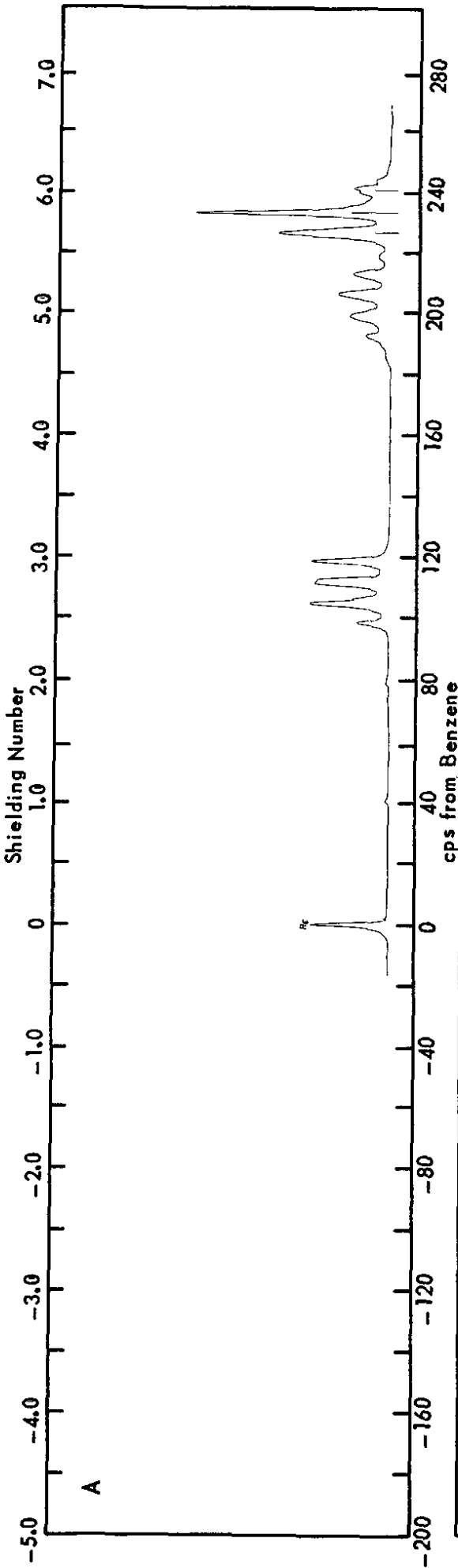
Sample		Ext. Reference		Instrument and Conditions	
(CH ₃ O) ₃ PO	Solvent	Benzene		Model: Varian V-4300B	Temperature: 26°C
	Conc.	Benzene, 5% v		Freq.: 40 Mc/sec	
Source and Purity Aldrich Chemical Co.		Resonance Position		Scan Rates, cps/sec:	
		-28.4 cps from Benzene		STD (Spectrum A): 0.99	
				Other (Spectrum B):	
				(Spectrum C):	



Sample		Ext. Reference		Instrument and Conditions	
		Benzene			
(CH ₃ -CH ₂ -O) ₃ PO β α		Int. Reference		Model: Varian V-4300B	
		Benzene, 5% v		Freq.: 40 Mc/sec	
Source and Purity Eastman (Prac.)		Resonance Position		Scan Rates, cps/sec:	
		-24.4 cps from Benzene		STD (Spectrum A): 0.99	
				Other (Spectrum B): 0.24	
				(Spectrum C): 0.24	
				Temperature: 26°C	



Sample		Ext. Reference		Instrument and Conditions	
(CCl ₃ -CH ₂ -O) ₃ PO		Benzene		Model: Varian V-4300B	
		Int. Reference		Freq.: 40 Mc/sec	
Source and Purity Aldrich Chemical Co.		Resonance Position cps from Benzene		Scan Rates, cps/sec: STD (Spectrum A): 1.08 Other (Spectrum B): (Spectrum C):	
				Temperature: 26°C	

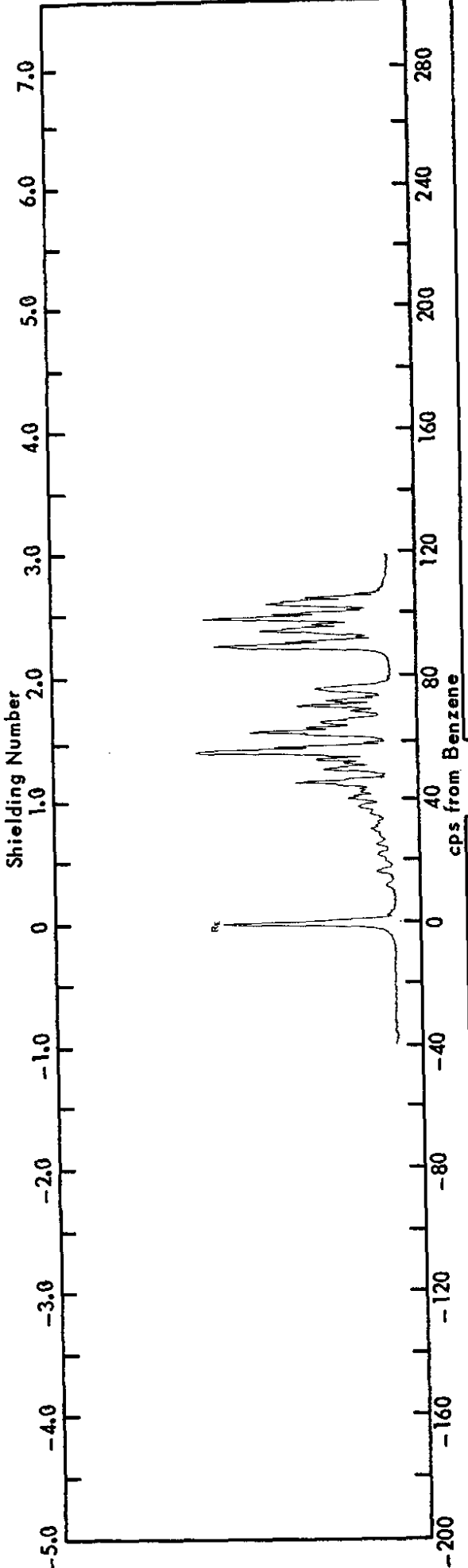


Sample		Solvent		Ext. Reference		Instrument and Conditions	
(CH ₃ -CH ₂ -CH ₂ -O) ₃ PO		Benzene		Benzene		Model: Varian V-4300B	
γ β α		Conc.		Benzene, 5% v		Freq.: 40 Mc/sec	
Source and Purity				Resonance Position		Scan Rates, cps/sec:	
Synthesis, molecular distillation				-24.4 cps from Benzene		STD (Spectrum A): 0.98	
						Other (Spectrum B): 0.47	
						(Spectrum C): 0.48	
						Temperature: 26°C	

Triallyl phosphate

Serial No. 5

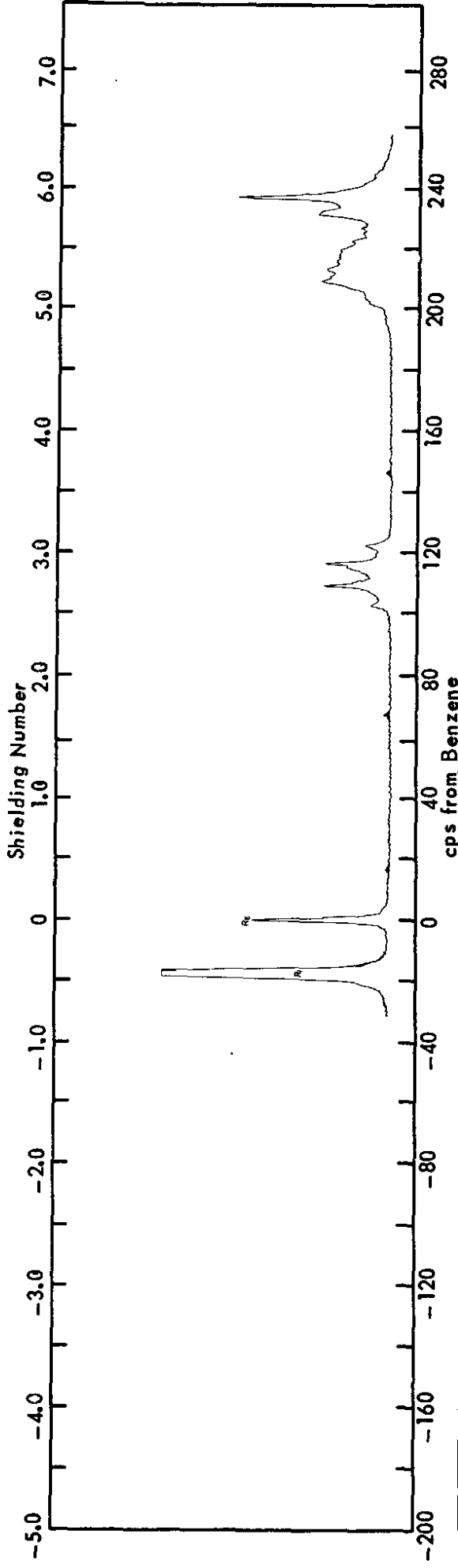
Proton Magnetic Resonance Spectral Data



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Sample		Ext. Reference		Instrument and Conditions	
(CH ₂ =CH-CH ₂ -O) ₃ PO		Benzene		Model: Varian V-4300B	
		Int. Reference		Freq.: 40 Mc/sec	
Source and Purity Aldrich Chemical Co.		Resonance Position cps from Benzene		Scan Rates, cps/sec: STD (Spectrum A): 1.06 Other (Spectrum B): (Spectrum C):	
				Temperature: 26°C	



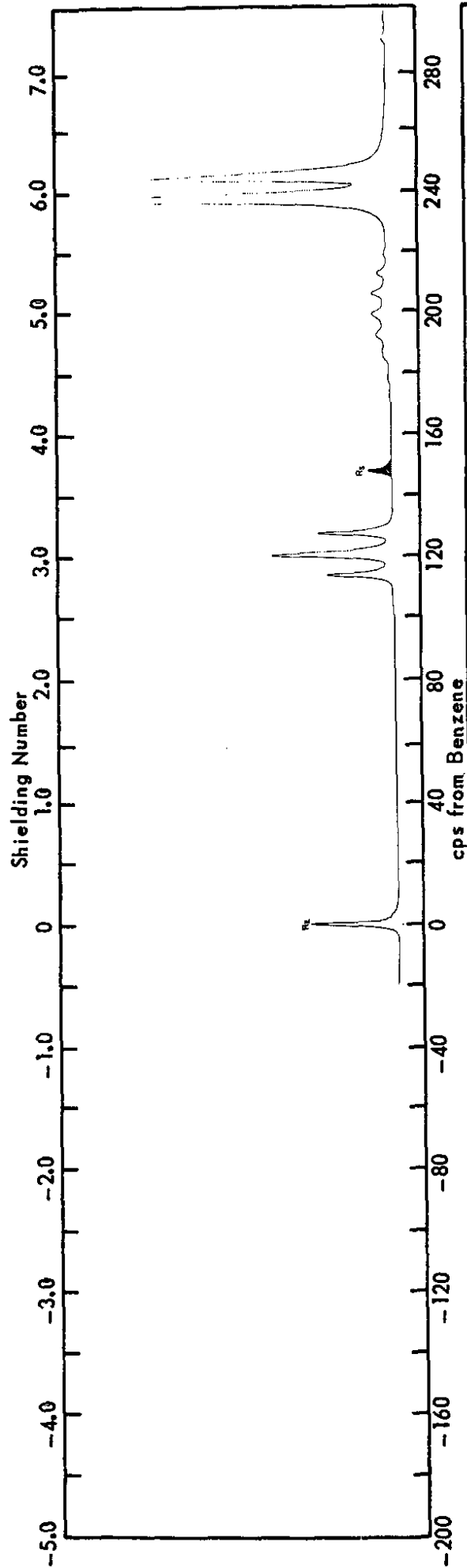
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Sample		Ext. Reference		Instrument and Conditions	
(CH ₃ -CH ₂ -CH ₂ -CH ₂ -O) ₃ PO	Solvent	Benzene		Model: Varian V-4300B Freq.: 40 Mc/sec Scan Rates, cps/sec: STD (Spectrum A): 0.96 Other (Spectrum B): (Spectrum C): Temperature: 26°C	
	Conc.	Benzene, 5% v			
Source and Purity Commercial Solvents Co.		Resonance Position -24.1 cps from Benzene			

Tri-iso-butyl phosphate

Proton Magnetic Resonance Spectral Data

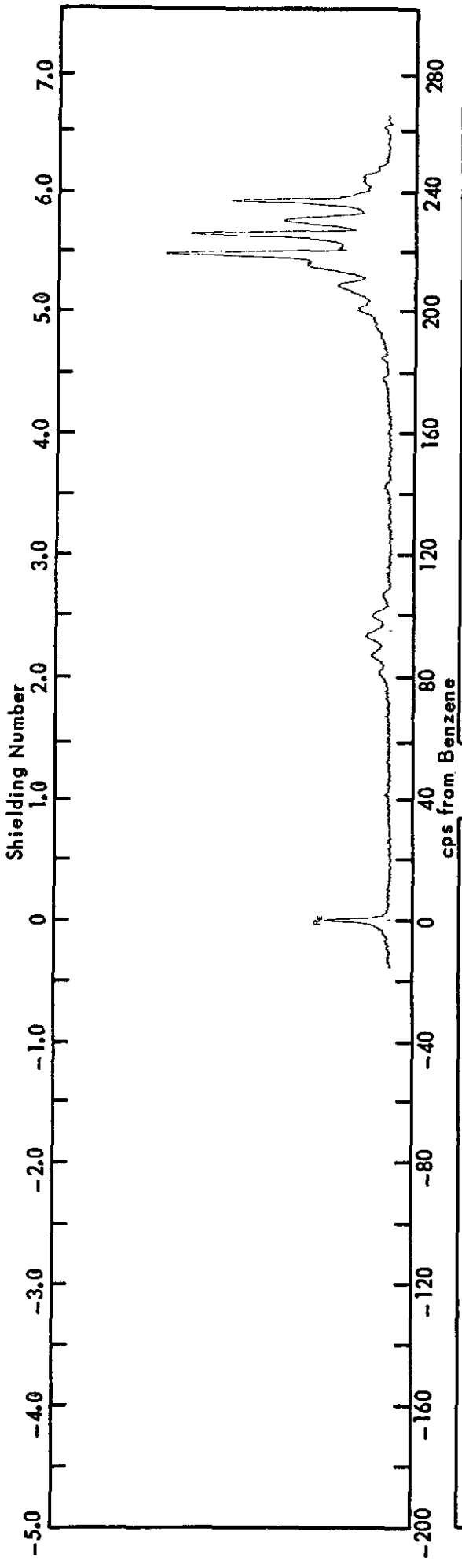
Serial No. 7



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Sample		Ext. Reference		Instrument and Conditions	
[(CH ₃) ₂ -CH-CH ₂ -O] ₃ PO	Solvent	Benzene		Model: Varian V-4300B	Temperature: 26°C
	Conc.	Benzene, 5%		Freq.: 40 Mc/sec	
Source and Purity Shea Chemical Co.	Resonance Position		Scan Rates, cps/sec:		
	-23.8 cps from Benzene		STD (Spectrum A): 0.99		
			Other (Spectrum B): (Spectrum C):		



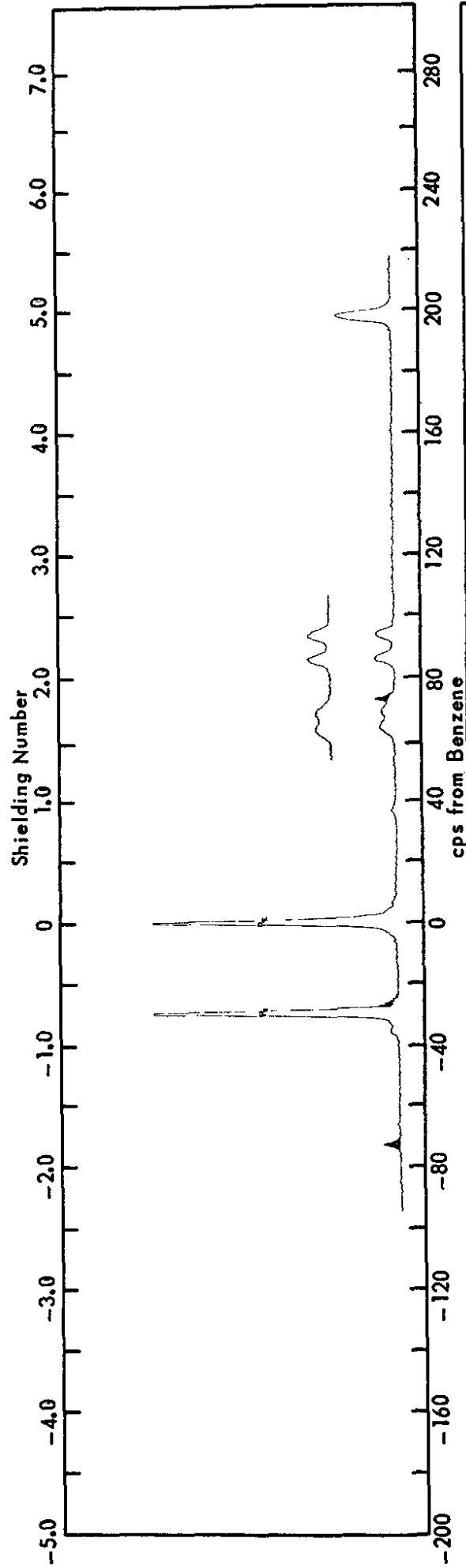
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Sample		Ext. Reference		Instrument and Conditions	
$(CH_3-CH_2-\underset{\substack{ \\ CH_3}}{CH}-O)_3PO$	Solvent	Benzene		Model: Varian V-4300B Freq.: 40 Mc/sec Scan Rates, cps/sec: STD (Spectrum A): 1.01 Other (Spectrum B): (Spectrum C): Temperature: 26°C	
	Conc.	Int. Reference			
Source and Purity Synthesis, crystallization		Resonance Position cps from Benzene			

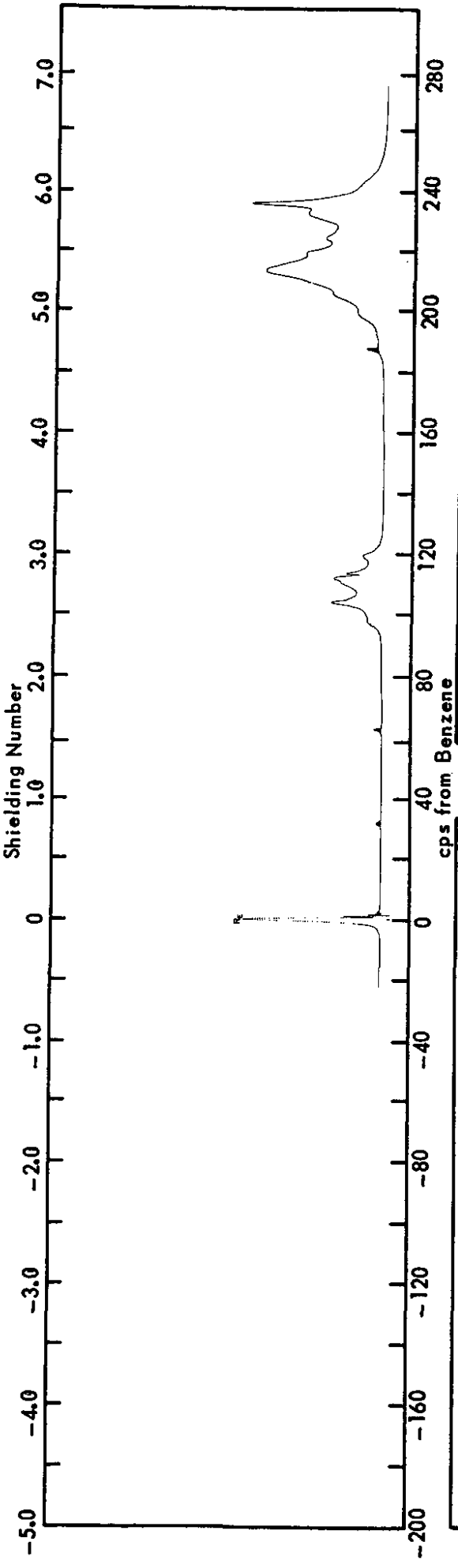
Trimethyl phosphate

Serial No. 9

Proton Magnetic Resonance Spectral Data



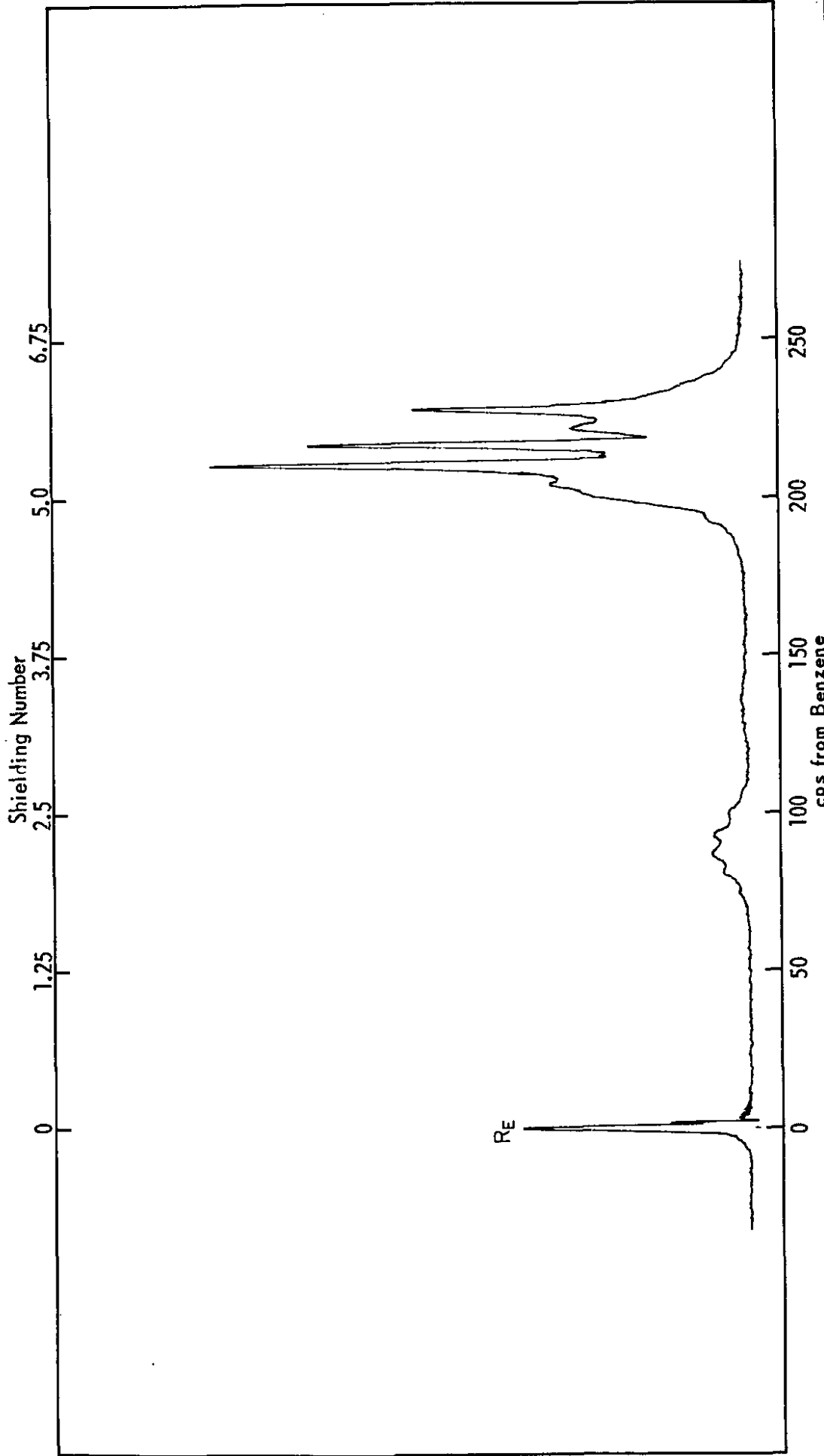
Sample		Ext. Reference		Instrument and Conditions	
$(\text{CH}_2=\underset{\text{CH}_3}{\text{C}}-\text{CH}_2-\text{O})_3\text{PO}$	Solvent	Benzene		Model: Varian V-4300B	Temperature: 26°C
	Conc.	Benzene, 75% v		Freq.: 40 Mc/sec	
Source and Purity Shea Chemical Co.	Resonance Position		Scan Rates, cps/sec:	STD (Spectrum A): 1.01	
	-28.44 cps from Benzene		Other (Spectrum B):		
			(Spectrum C):		



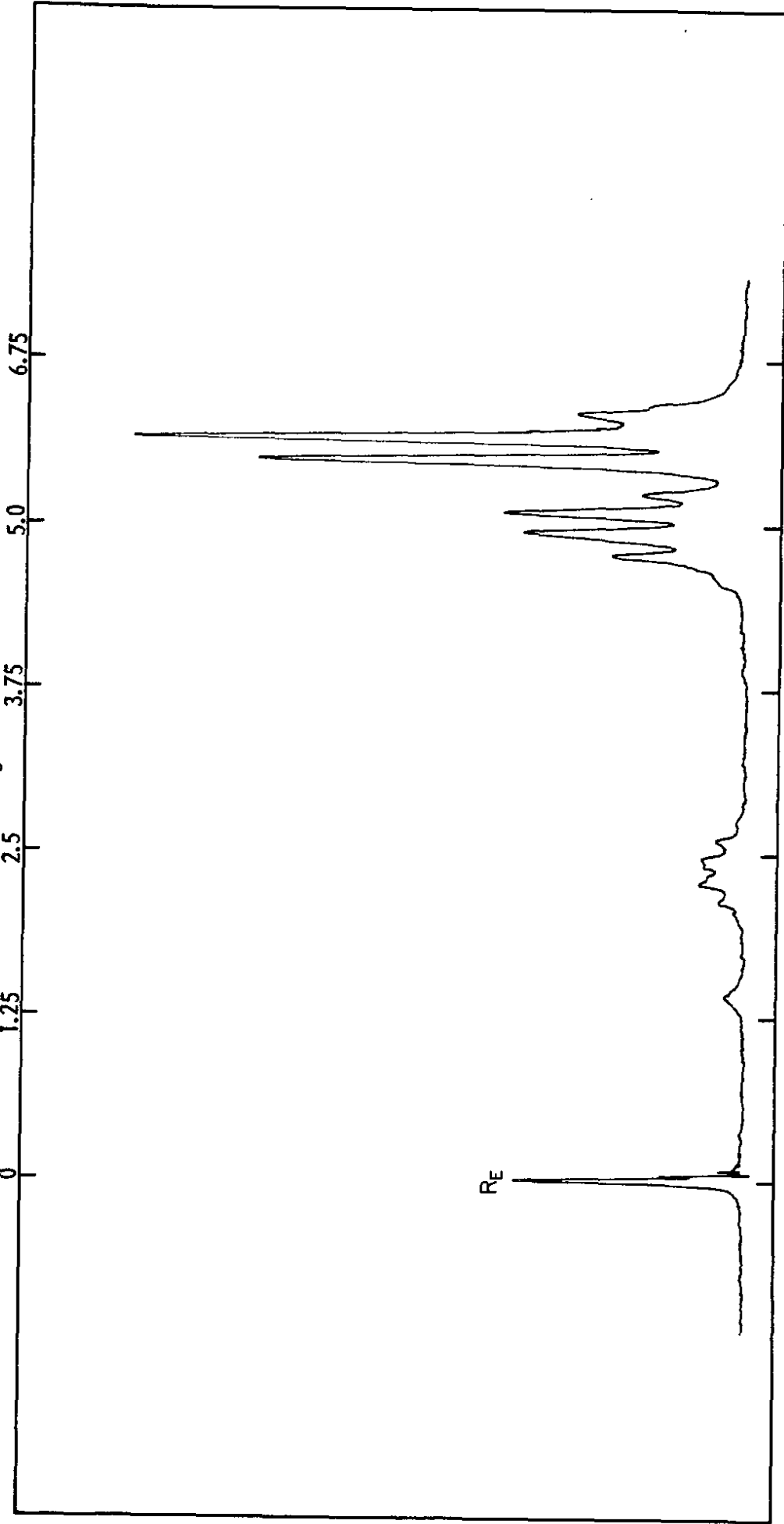
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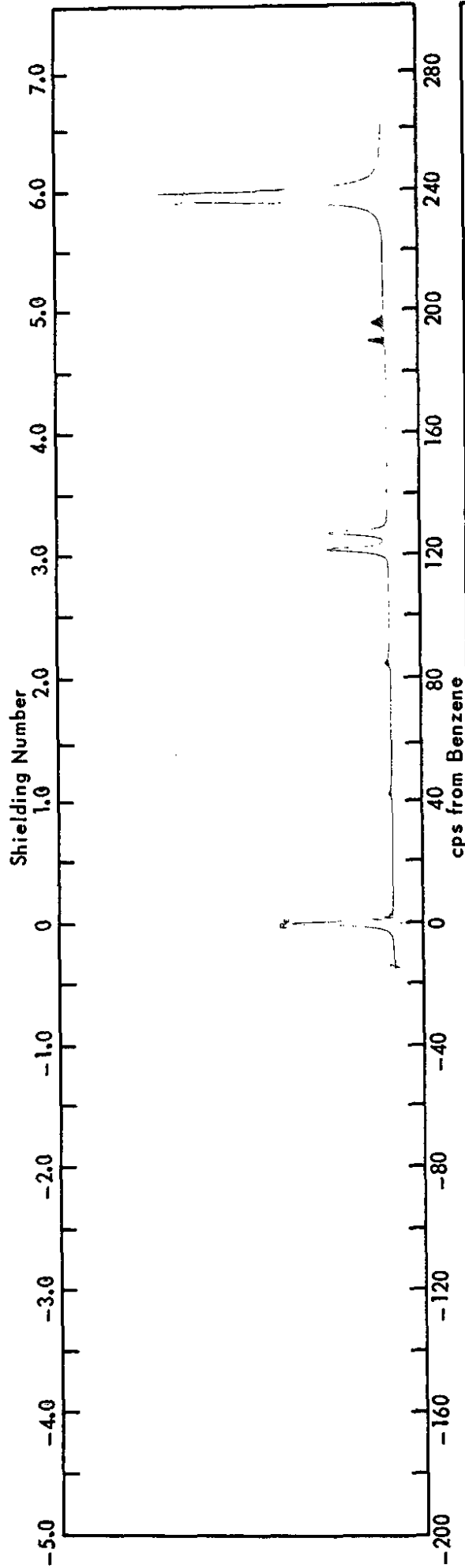
Sample		Ext. Reference		Instrument and Conditions	
(CH ₃ -CH ₂ -CH ₂ -CH ₂ -CH ₂ -O) ₃ PO		Benzene		Model: Varian V-4300B Freq.: 40 Mc/sec Scan Rates, cps/sec: STD (Spectrum A): 0.99 Other (Spectrum B): (Spectrum C): Temperature: 26°C	
		Benzene, 5% v			
Source and Purity Synthesis, molecular distillation		Resonance Position -25.0 cps from Benzene			



Sample $(\text{CH}_3\text{-CH}_2\text{-CH}_2\text{-CH}_2\text{-CH}\cdot\text{O})_3\text{PO}$ CH_3		Ext. Reference		Instrument and Conditions Model: Varian V-4300B Freq.: 40 Mc/sec Scan Rates, cps/sec: STD (Spectrum A): 2.44 Other (Spectrum B): (Spectrum C): Temperature: 26°C
		Benzene		
Solvent		Int. Reference		
Conc.		Resonance Position		
Source and Purity		cps from Benzene		
Synthesis, crystallized		cps from Benzene		



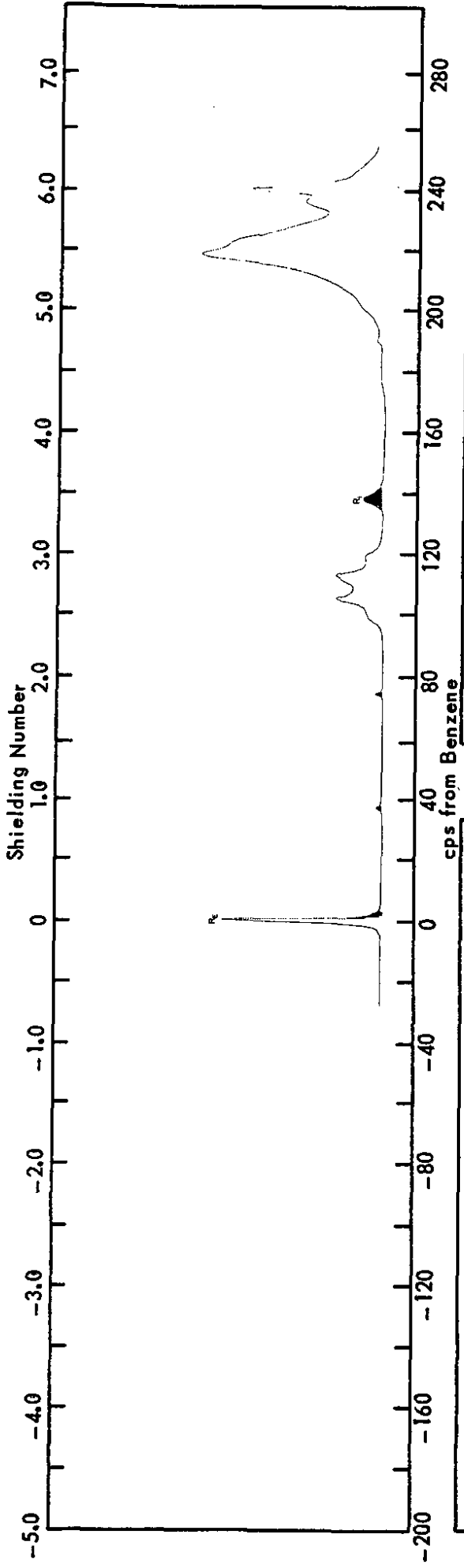
Sample		Instrument and Conditions	
[(CH ₃ -CH ₂) ₂ CH-O] ₃ PO	Solvent	Model: Varian V-4300B	
	Conc.	Freq: 40 Mc/sec	
Source and Purity Synthesis, crystallized		Scan Rates, cps/sec:	
		STD (Spectrum A): 2.44	
		Other (Spectrum B): (Spectrum C):	
		Temperature: 26°C	
		Ext. Reference Benzene	
		Int. Reference	
		Resonance Position cps from Benzene	



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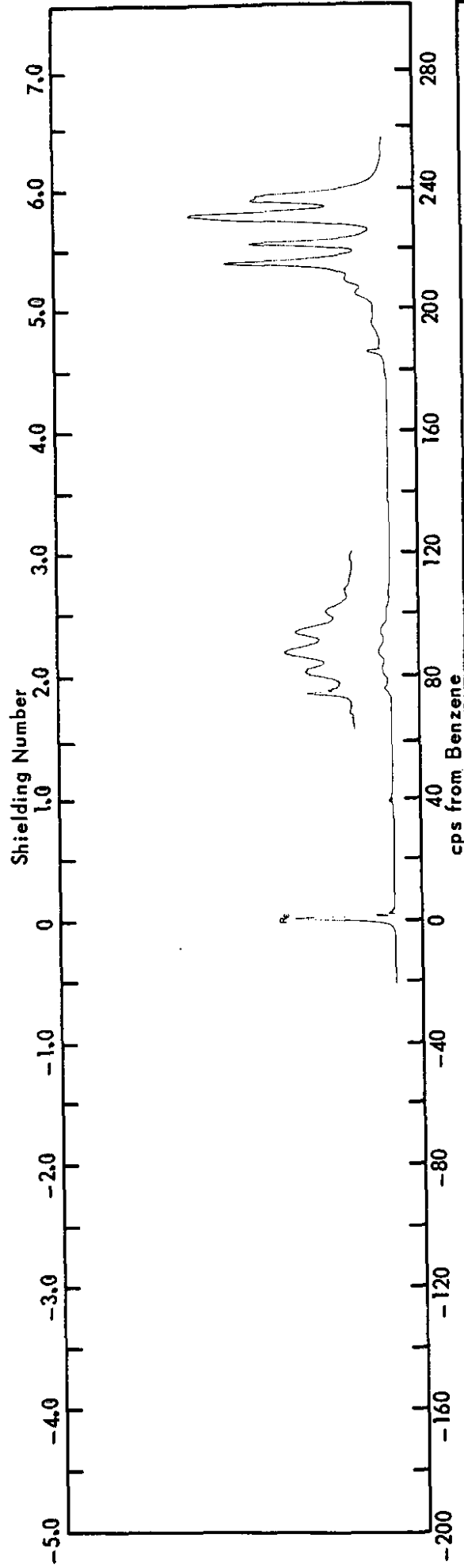
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Sample		Ext. Reference		Instrument and Conditions	
$\begin{array}{c} \text{CH}_3 \\ \\ (\text{CH}_3 - \text{C} - \text{CH}_2 - \text{O})_3 \text{PO} \\ \\ \text{CH}_3 \end{array}$		Benzene		Model: Varian V-4300B	
		Benzene, 5% v		Freq.: 40 Mc/sec	
Source and Purity E. I. du Pont de Nemours & Co.		Resonance Position -24.9 cps from Benzene		Scan Rates, cps/sec: STD (Spectrum A): 1.04 Other (Spectrum B): (Spectrum C):	
				Temperature: 26°C	



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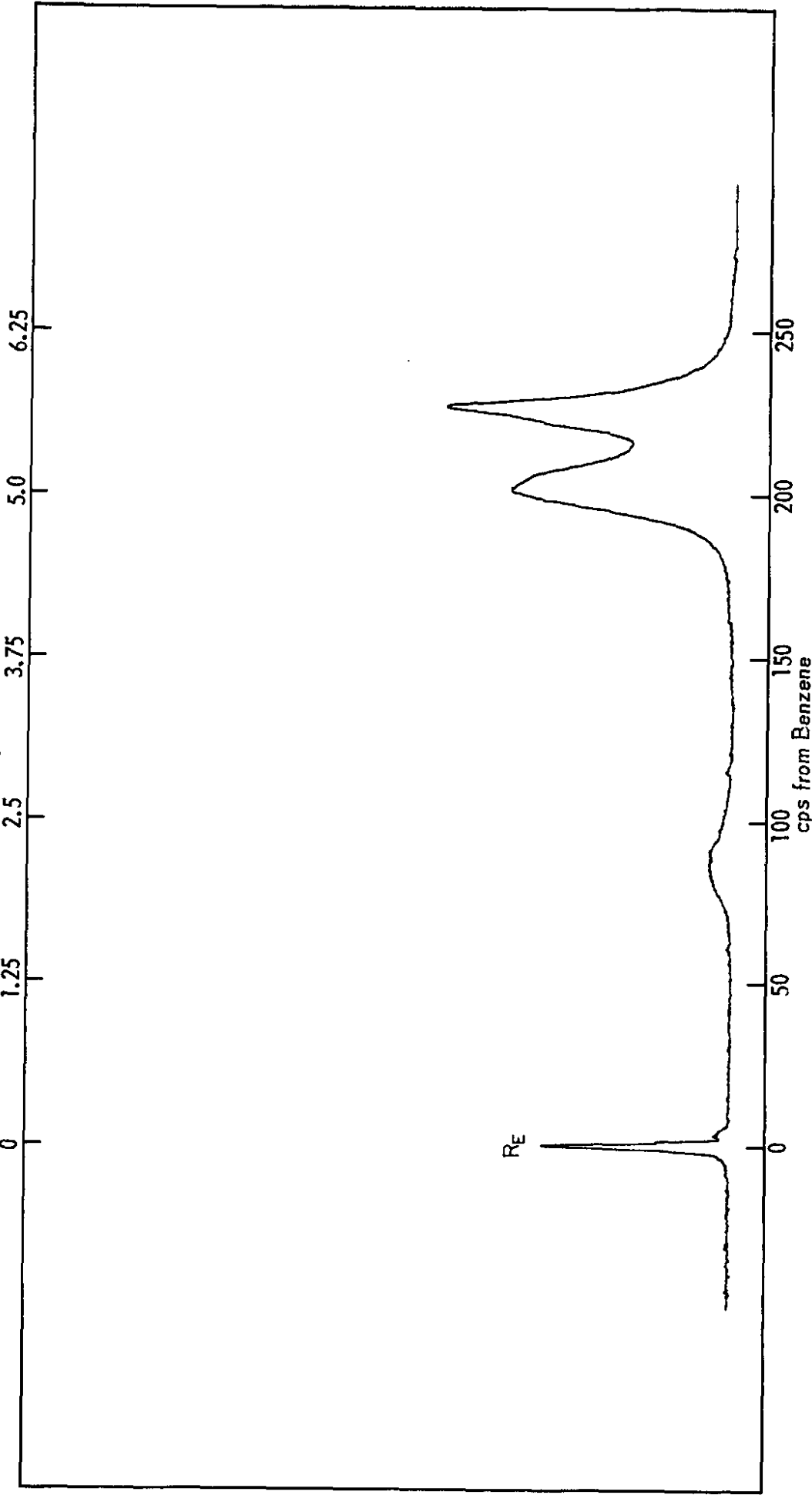
Sample		Ext. Reference		Instrument and Conditions	
$[\text{CH}_3 \cdot (\text{CH}_2)_5 \cdot \text{O}]_3 \text{PO}$		Benzene		Model: Varian V-4300B Freq.: 40 Mc/sec Scan Rates, cps/sec: STD (Spectrum A): 1.01 Other (Spectrum B): (Spectrum C): Temperature: 26°C	
		Benzene, 5% v			
Source and Purity Synthesis, crystallized		Resonance Position -24.5 cps from Benzene			



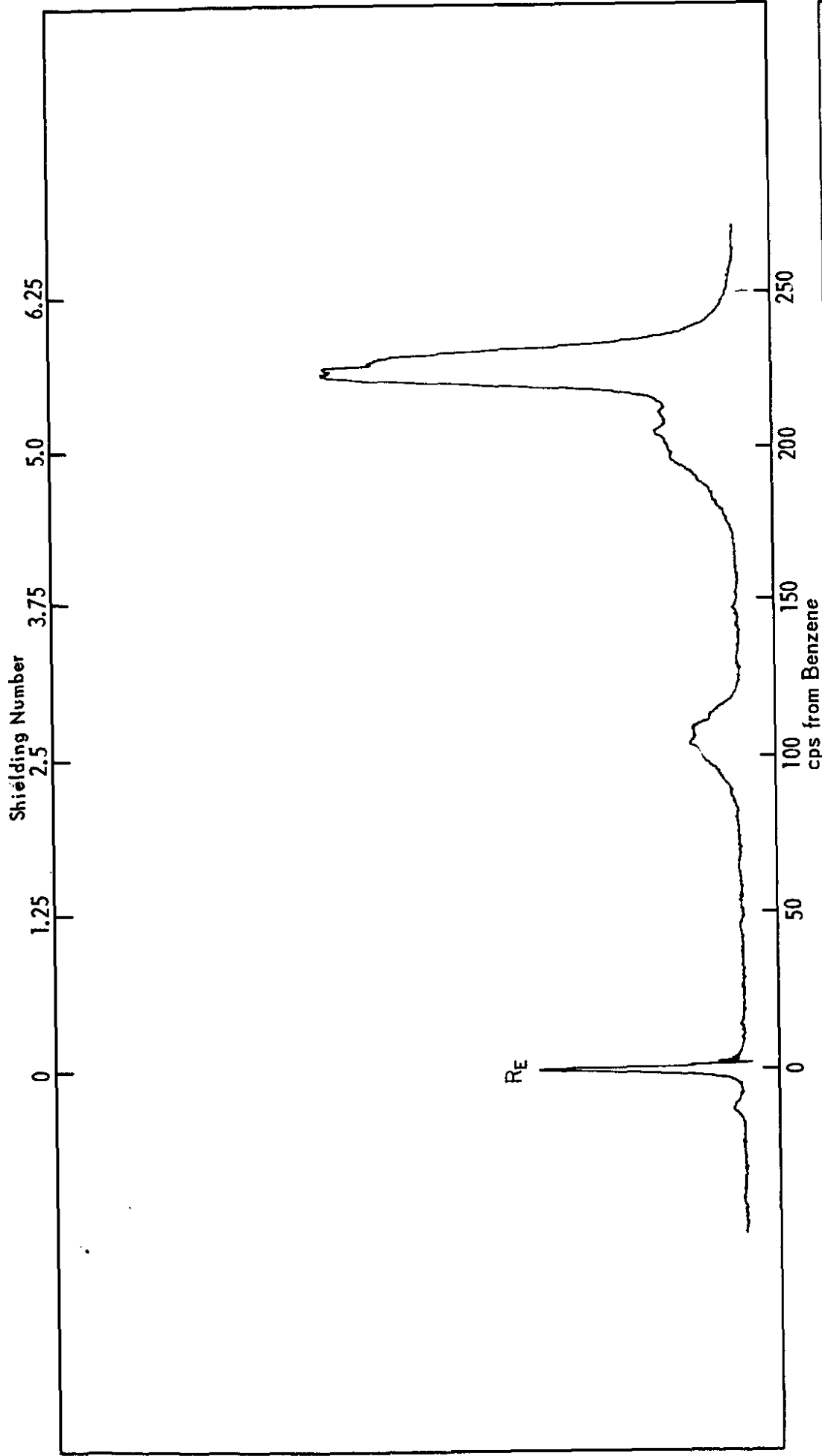
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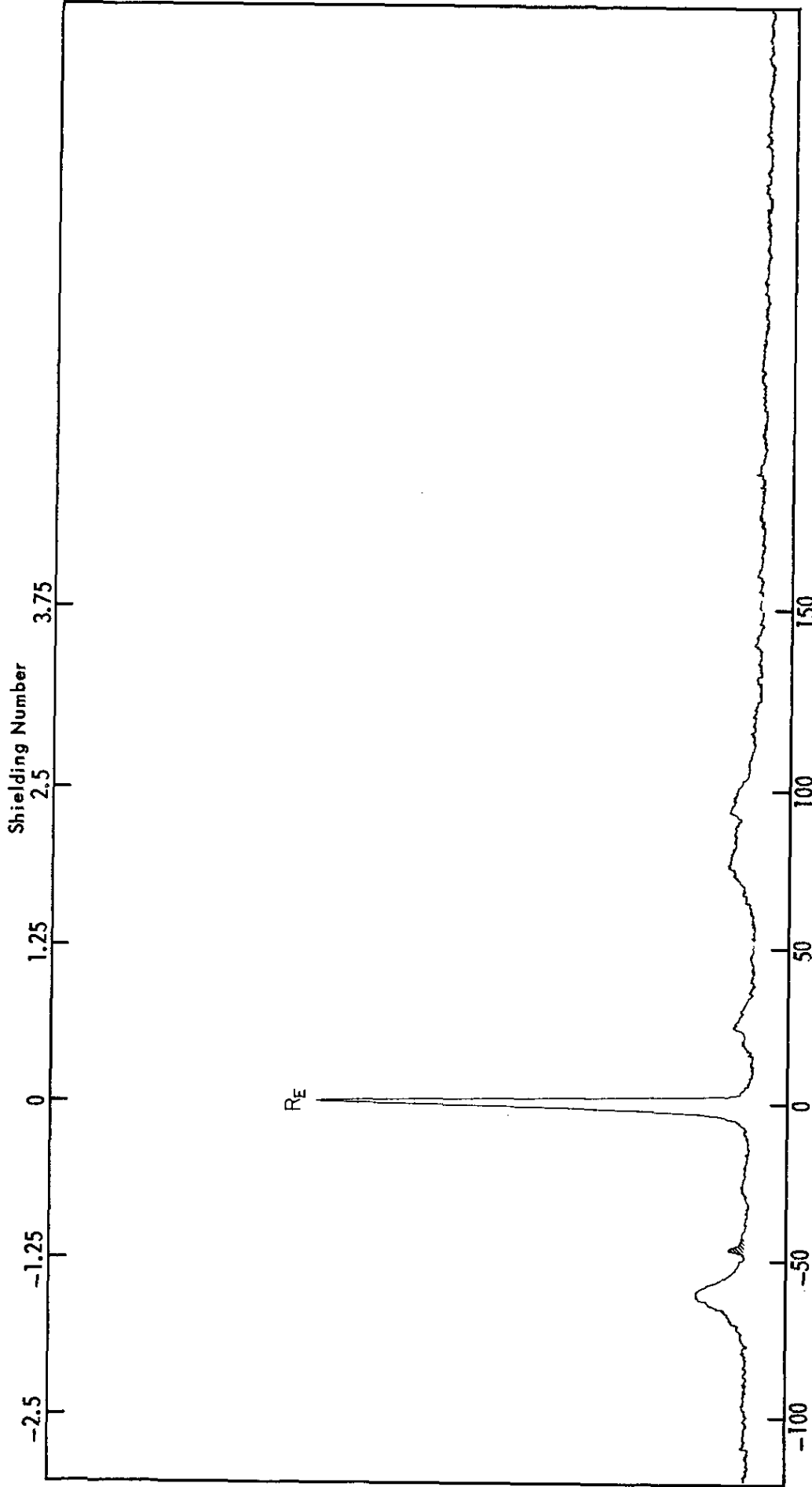
Sample		Ext. Reference		Instrument and Conditions	
$\begin{array}{c} \text{CH}_3 \\ \\ (\text{CH}_3-\text{CH}-\text{CH}_2-\text{CH}-\text{O})_3 \text{PO} \\ \\ \text{CH}_3 \end{array}$	Solvent	Benzene		Model: Varian V-4300B	
	Conc.	Benzene, 5%		Freq: 40 Mc/sec	
Source and Purity Synthesis, molecular distillation		Resonance Position		Scan Rates, cps/sec:	
		-25.0 cps from Benzene		STD (Spectrum A): 1.01	
				Other (Spectrum B):	
				(Spectrum C):	
				Temperature: 26°C	



Sample		Ext. Reference		Instrument and Conditions	
$[(\text{CH}_3 - \text{CH}_2 - \text{CH}_2)_2 - \text{CH} \cdot \text{O}]_3 \text{PO}$	Solvent	Benzene		Model: Varian V-4300B	
	Conc.	Int. Reference		Freq.: 40 Mc/sec	
Source and Purity Synthesis, crystallized	Resonance Position cps from Benzene		Scan Rates, cps/sec: 2.46		STD (Spectrum A): Other (Spectrum B): (Spectrum C): Temperature: 26°C



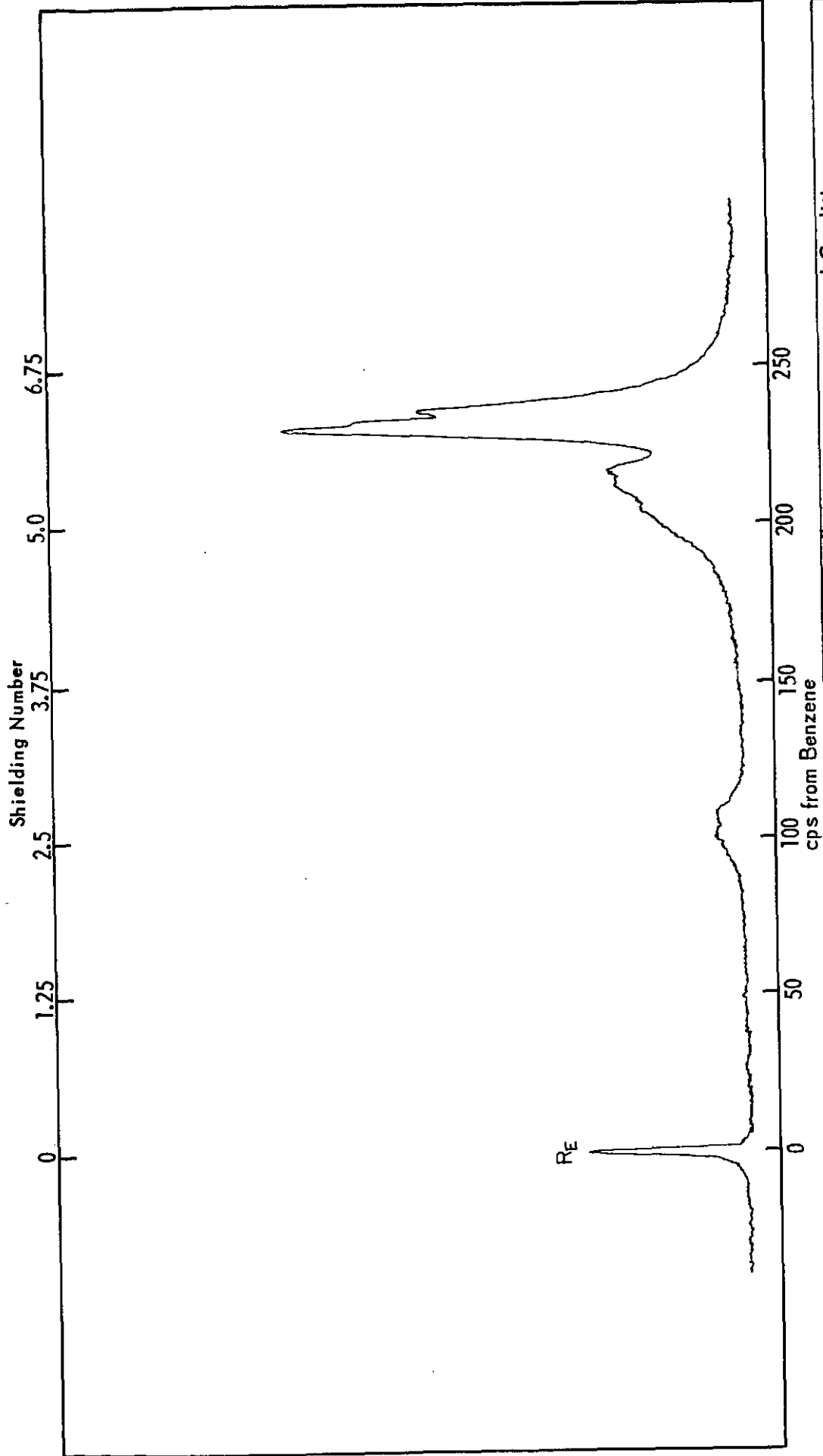
Sample		Ext. Reference		Instrument and Conditions	
$\left\{ \left[(\text{CH}_3)_2\text{CH} \right]_2 \text{CH}-\text{O} \right\}_3 \text{PO}$	Solvent	Benzene		Model: Varian V-4300B	
	Conc.	Int. Reference		Freq.: 40 Mc/sec	
Source and Purity Synthesis, crystallized		Resonance Position cps from Benzene		Scan Rates, cps/sec: STD (Spectrum A): 2.45 Other (Spectrum B): (Spectrum C):	
				Temperature: 26°C	



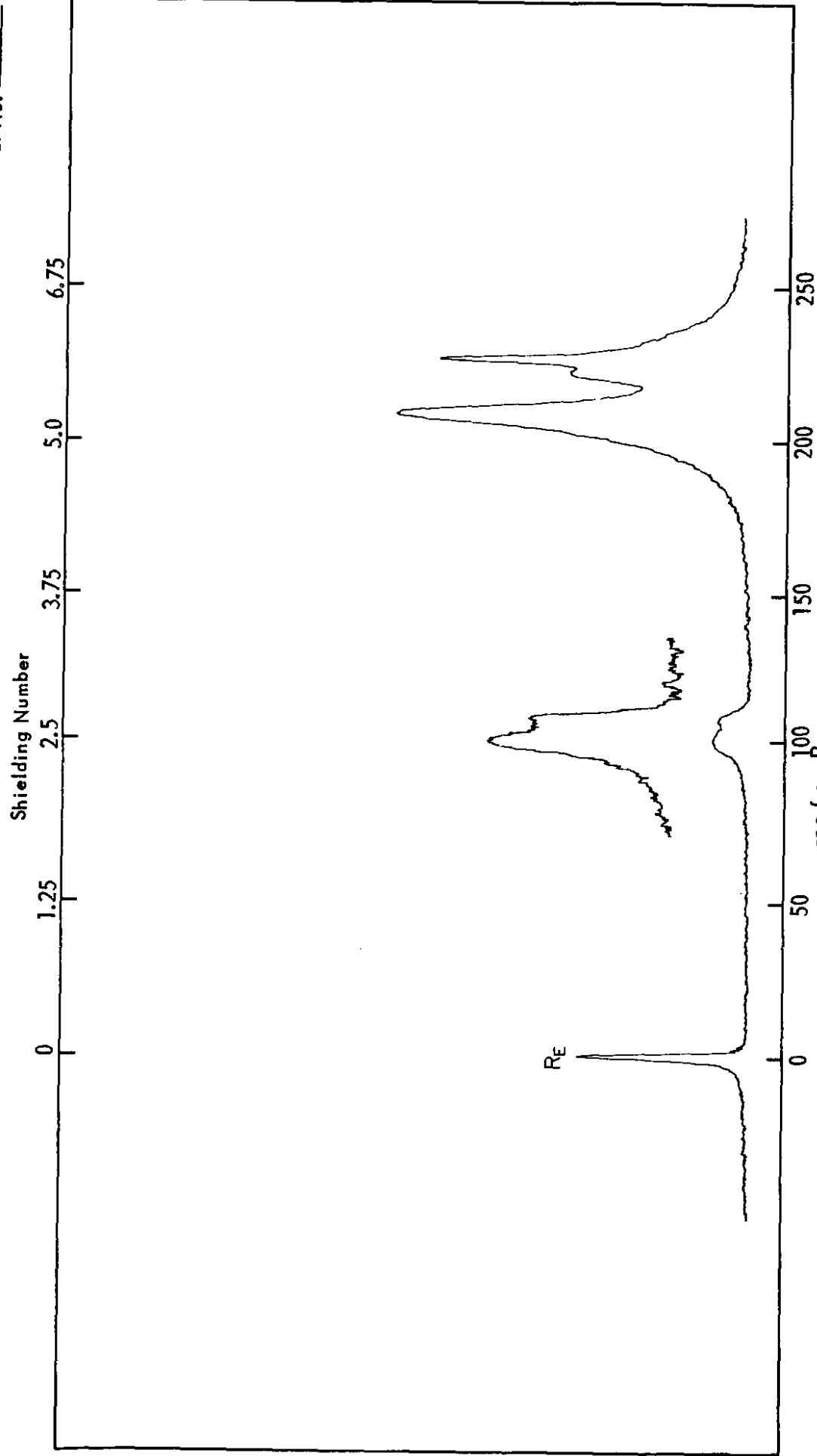
Sample $[\text{CHF}_2-(\text{CF}_2)_5-\text{CH}_2\text{F}-\text{O}]_3\text{PO}$		Solvent		Instrument and Conditions Model: Varian V-4300B Freq: 40 Mc/sec Scan Rates, cps/sec: STD (Spectrum A): 2.36 Other (Spectrum B): (Spectrum C): Temperature: 26°C	
		Conc.			
Source and Purity		Ext. Reference Benzene		Resonance Position cps from Benzene	
		Int. Reference			

Proton Magnetic Resonance Spectral Data

Tri-iso-octyl phosphate

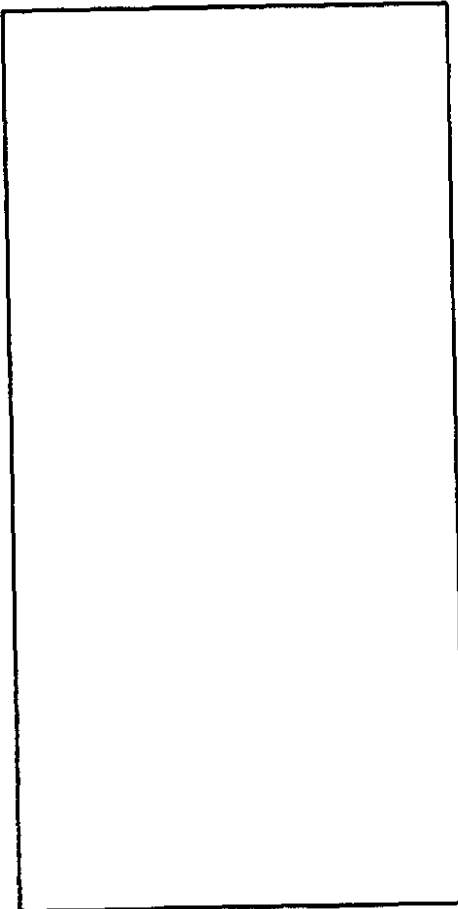
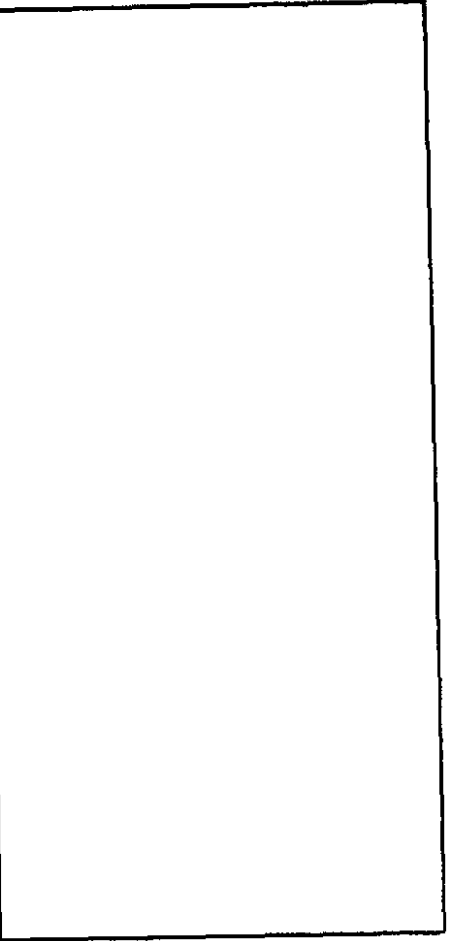
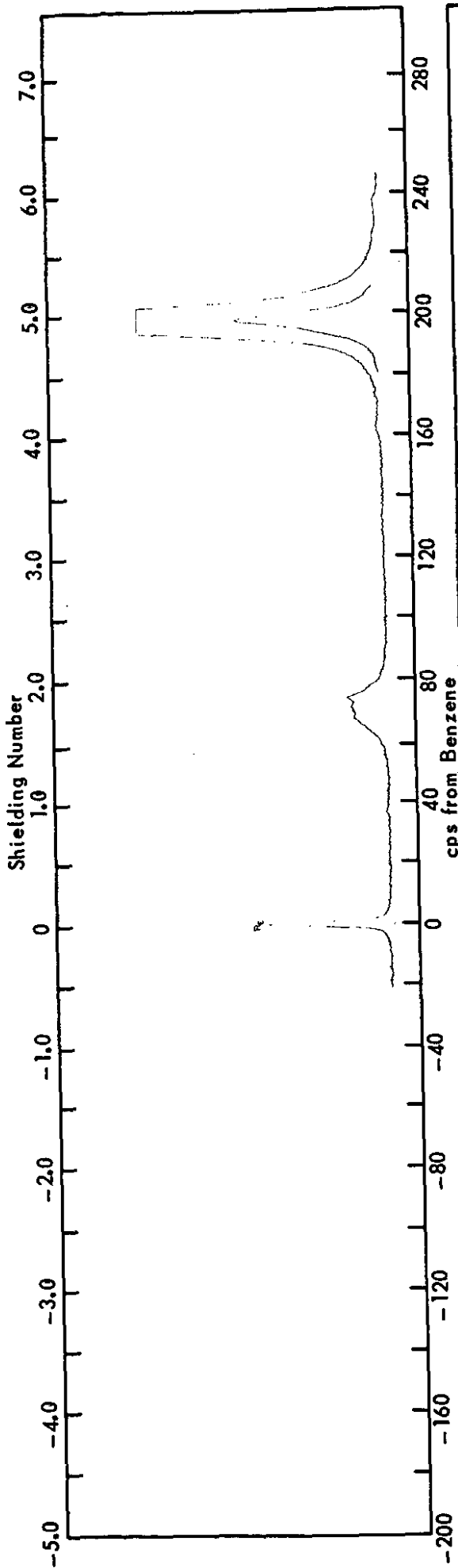


Sample		Ext. Reference		Instrument and Conditions	
$[(CH_3)_2-CH-(CH_2)_5-O]_3 PO$ Source and Purity Synthesis, crystallized		Solvent	Benzene	Model: Varian V-4300B	
		Conc.	Int. Reference	Freq: 40 Mc/sec	
			Resonance Position cps from Benzene	Scan Rates, cps/sec: STD (Spectrum A): 2.54 Other (Spectrum B): (Spectrum C):	
				Temperature: 26°C	

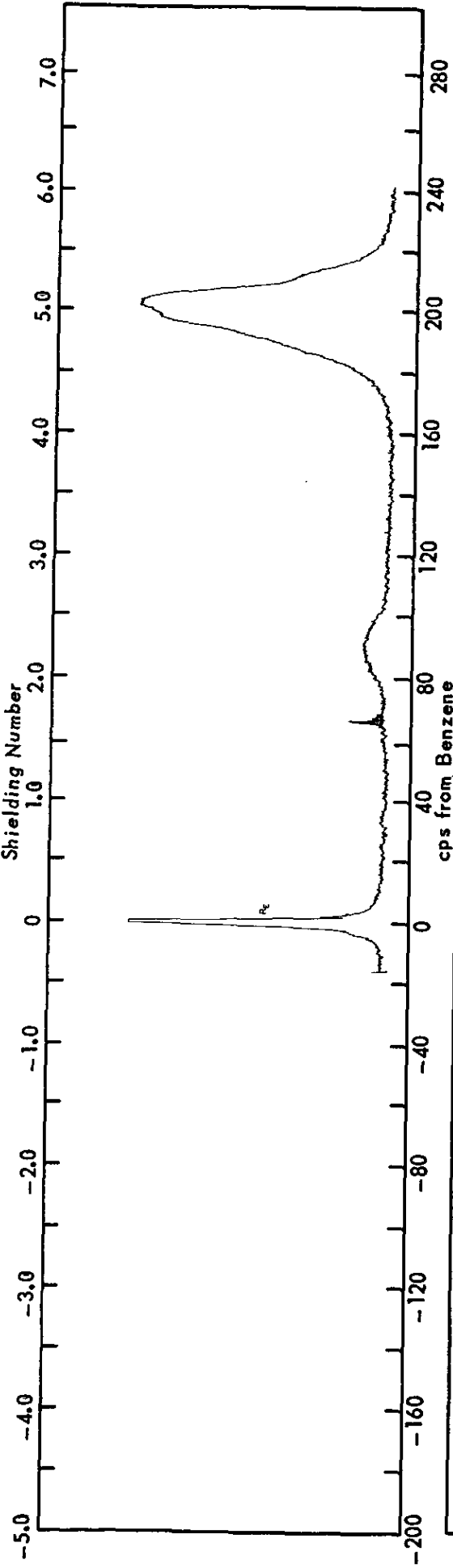


Sample $[\text{CH}_3 \cdot \text{CH}_2 \cdot \text{CH}_2 \cdot \text{CH}_2 \cdot \text{CH}(\text{C}_2\text{H}_5) \cdot \text{O}]_3 \text{PO}$		Solvent		Instrument and Conditions Model: Varian V-4300B Freq: 40 Mc/sec Scan Rates, cps/sec: STD (Spectrum A): 2.48 Other (Spectrum B): (Spectrum C): Temperature: 26°C
		Conc.		
Source and Purity Monsanto Chemical Co.		Ext. Reference Benzene		
		Int. Reference		
		Resonance Position cps from Benzene		

Proton Magnetic Resonance Spectral Data



Sample		Ext. Reference		Instrument and Conditions
$\left(\begin{array}{c} \text{CH}_2 \cdot \text{CH}_2 \\ \\ \text{CH}_2 \cdot \text{CH}_2 \end{array} \right)_3 \text{CH} \cdot \text{O} \text{ PO}_3$	Solvent	Benzene	Model: Varian V-4300B Freq.: 40 Mc/sec Scan Rates, cps/sec: STD (Spectrum A): 1.03 Other (Spectrum B): (Spectrum C): Temperature:	
	Conc.	Benzene, 5% v		
Source and Purity		Resonance Position		
Synthesis, crystallized		-30.3 cps from Benzene		

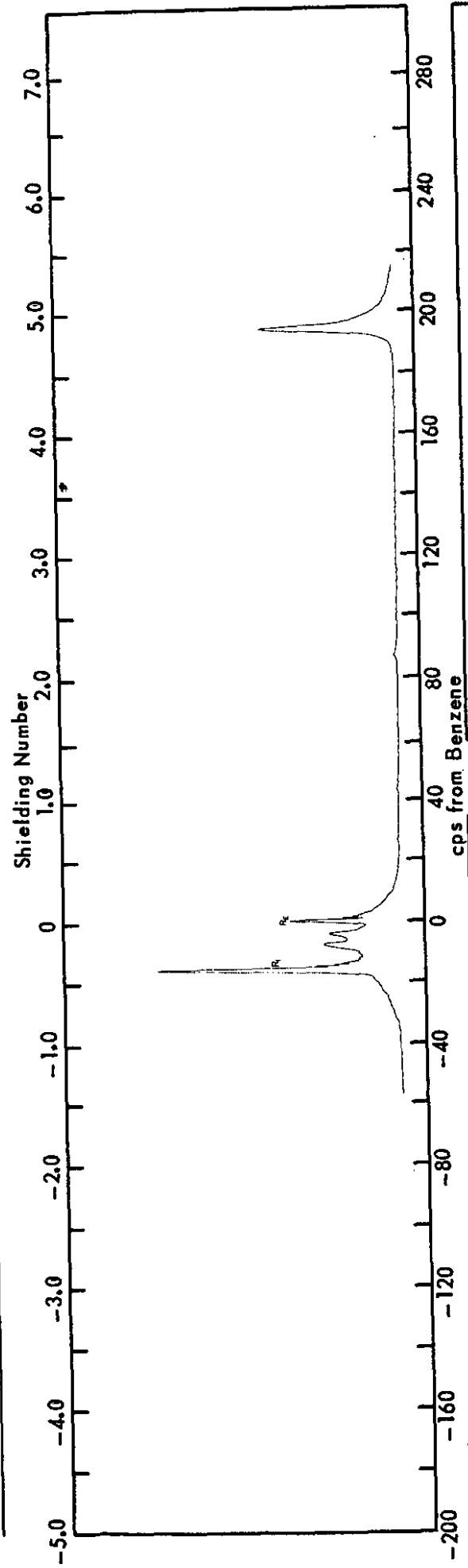


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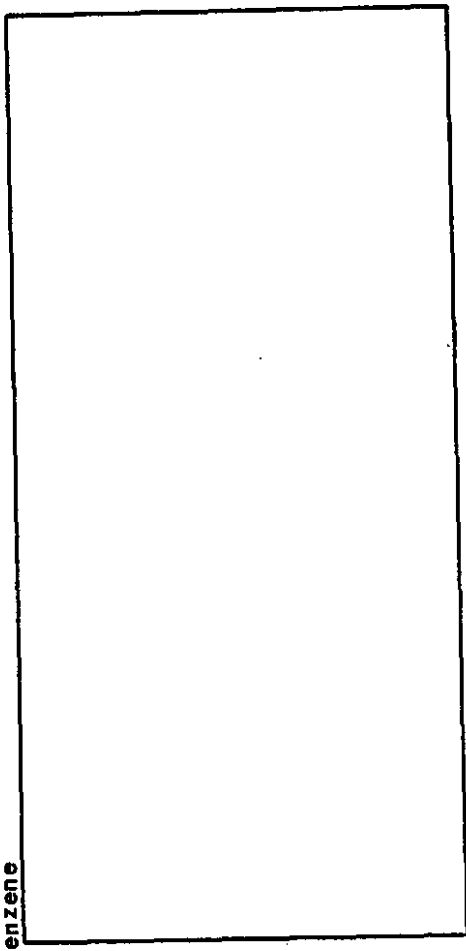
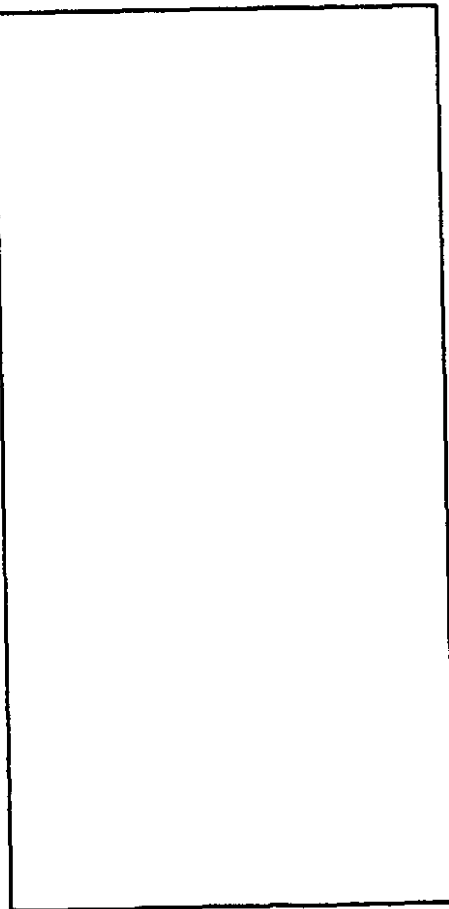
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Sample $\left(\begin{array}{c} \text{CH}_2\text{-CH}_2 \\ \text{CH}_2\text{-CH}_2 \\ \text{CH-O} \end{array} \right)_3 \text{PO}$		Ext. Reference		Instrument and Conditions Model: Varian V-4300B Freq.: 40 Mc/sec Scan Rates, cps/sec: STD (Spectrum A): 1.02 Other (Spectrum B): (Spectrum C): Temperature: 26°C
		Benzene		
Solvent		Int. Reference		
Conc.		Benzene, 5% v		
Source and Purity		Resonance Position		
Synthesis, crystallized		-27.2 cps from Benzene		

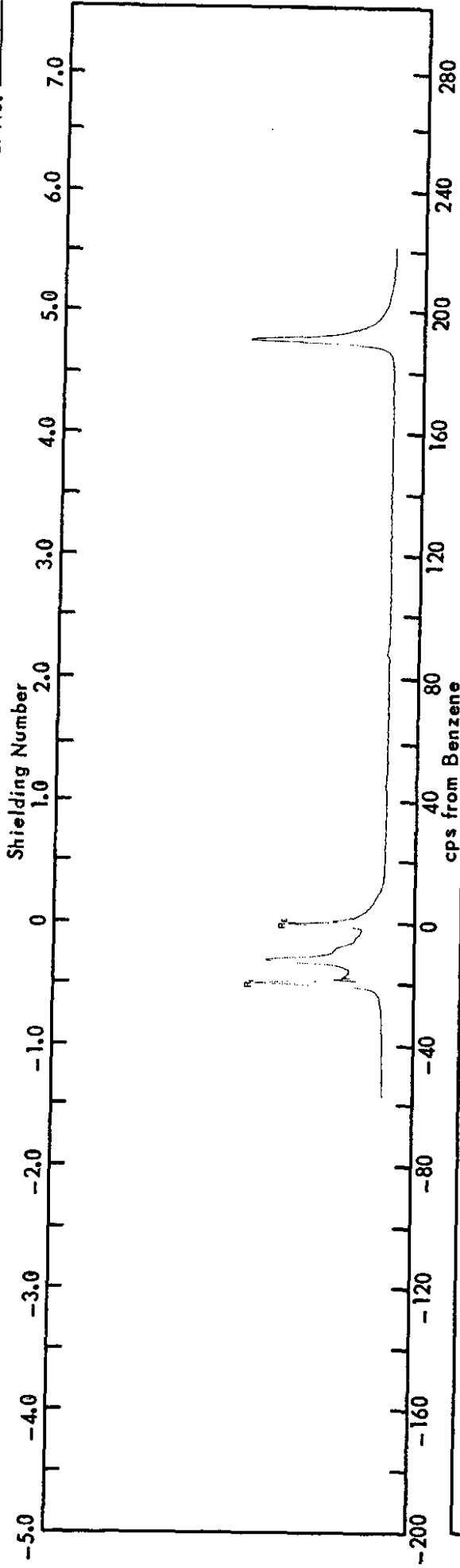
Proton Magnetic Resonance Spectral Data



Tri-*o*-tolyl phosphate

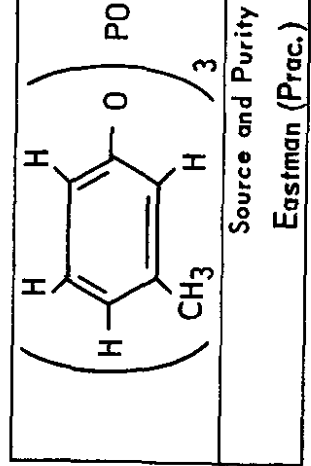


Sample		Ext. Reference		Instrument and Conditions	
<chem>Cc1cc(Oc2cc(C)cc2)c(O)c1</chem> Source and Purity Eastman (Prac.)	Solvent	Benzene		Model:	Varian V-4300B
	Conc.	Benzene, 8% v		Freq.:	40 Mc/sec
		Resonance Position		Scan Rates, cps/sec:	STD (Spectrum A): 1.05 Other (Spectrum B): (Spectrum C):
		-15.5 cps from Benzene		Temperature:	26°C



Sample	
Solvent	CCl ₄
Conc.	50% v

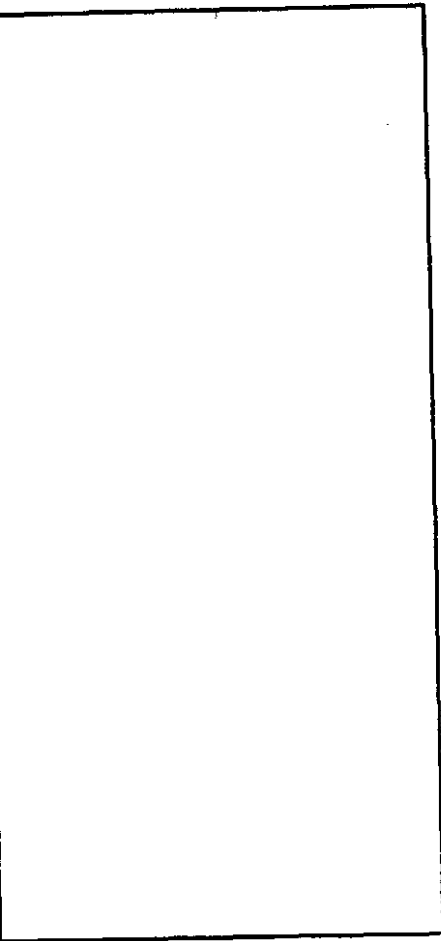
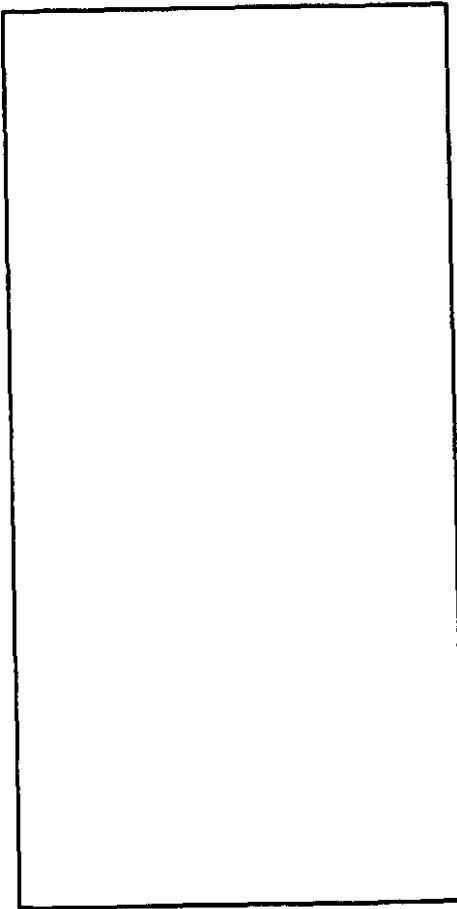
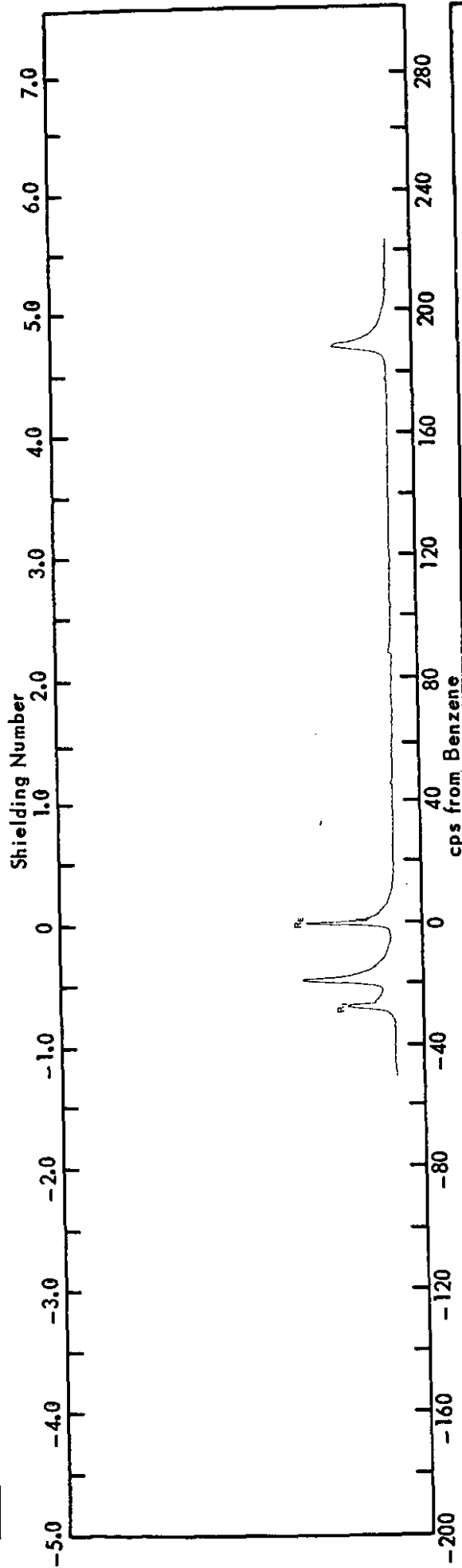
Ext. Reference	Benzene
Int. Reference	Benzene, 5% v
Resonance Position	-18.7 cps from Benzene



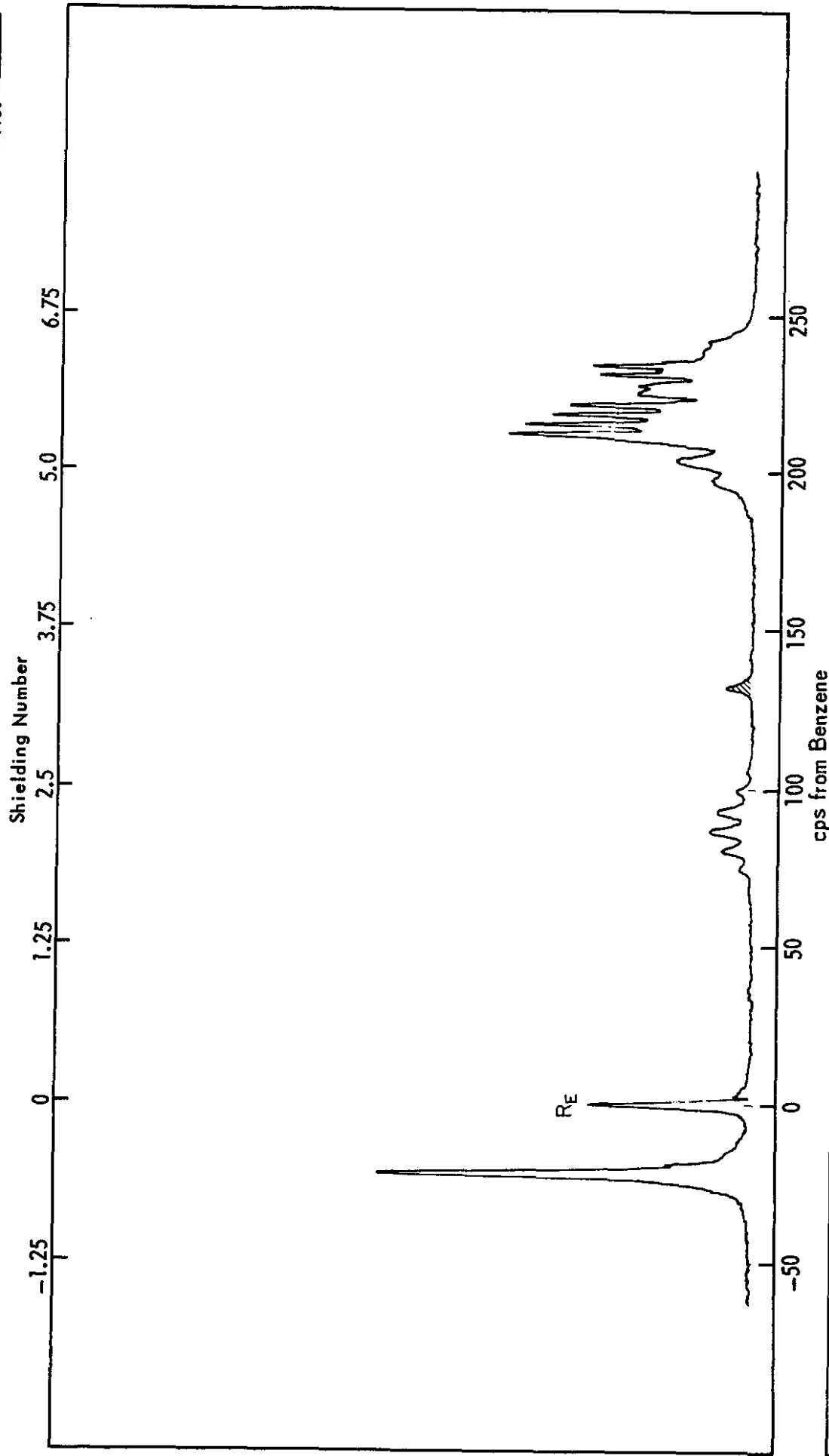
Instrument and Conditions
Model: Varian V-4300B
Freq.: 40 Mc/sec
Scan Rates, cps/sec: STD (Spectrum A): 1.02
Other (Spectrum B): (Spectrum C):
Temperature: 26°C

Proton Magnetic Resonance Spectral Data

Tri-p-tolyl phosphate



<chem>Cc1ccc(Oc2ccc(C)cc2)cc1</chem>		Sample	Instrument and Conditions	
		Solvent	Model: Varian V-4300B	
Source and Purity Eastman - recrystallized		Conc.	Freq.: 40 Mc/sec	
		Saturated	Scan Rates, cps/sec: STD (Spectrum A): 1.07 Other (Spectrum B): (Spectrum C):	
Ext. Reference		Temperature: 26°C		
Benzene		Resonance Position		
Int. Reference		-26.2 cps from Benzene		
Benzene, 3% v				

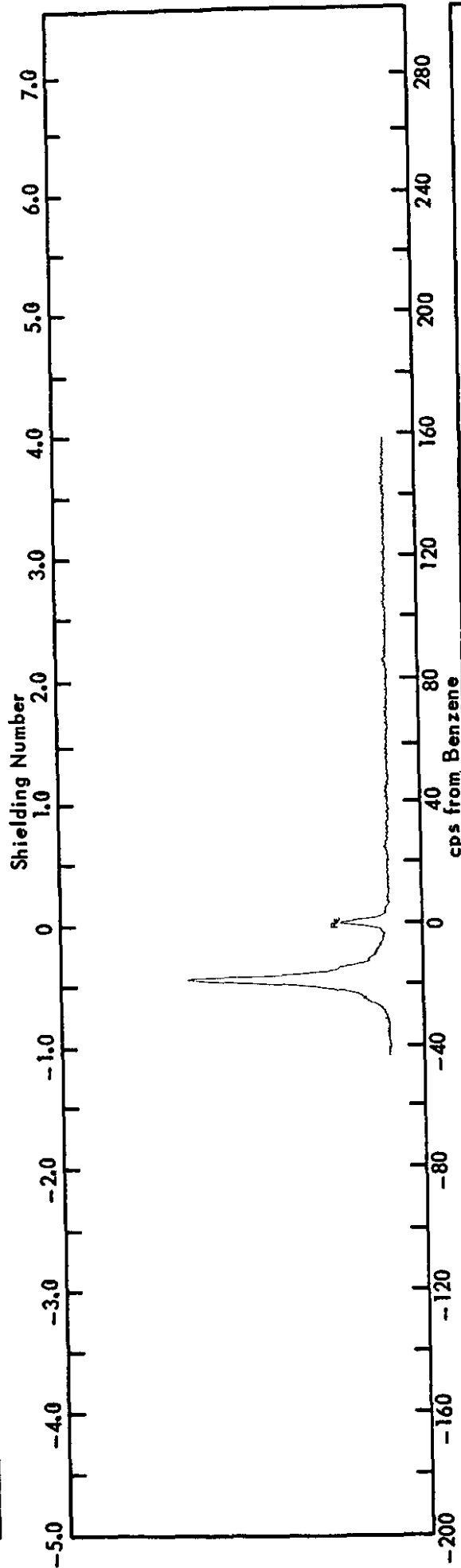


<p>Sample</p> $\left(\begin{array}{c} \text{CH}_3 \cdot \text{CH}_2 \cdot \text{CH} \cdot \text{O} \cdot \\ \\ \text{CH}_3 \end{array} \right)_2 \left(\begin{array}{c} \text{H} \\ \\ \text{C}_6\text{H}_4 \\ \\ \text{O} \cdot \text{PO} \end{array} \right)$	<p>Ext. Reference Benzene</p> <p>Int. Reference</p>	<p>Instrument and Conditions.</p> <p>Model: Varian V-4300B Freq.: 40 Mc/sec Scan Rates, cps/sec: STD (Spectrum A): 2.43 Other (Spectrum B): (Spectrum C): Temperature: 26°C</p>
<p>Source and Purity</p> <p>Monsanto Chemical Co.</p>	<p>Resonance Position cps from Benzene</p>	

Triphenyl phosphate

Serial No. 27

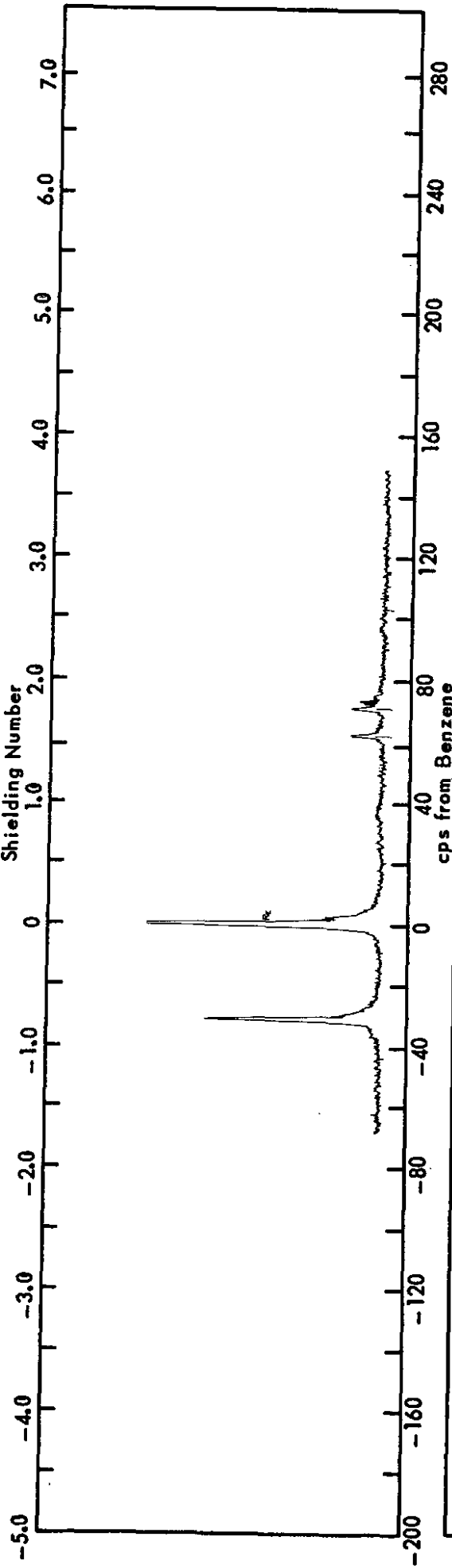
Proton Magnetic Resonance Spectral Data



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<p>Sample</p> <chem>O=P(O)(c1ccccc1)c2ccccc2</chem> <p>Source and Purity Eastman (Prac.)</p>	<p>Solvent CCl₄ Conc. Saturated</p>	<p>Ext. Reference Benzene</p> <p>Int. Reference</p> <p>Resonance Position cps from Benzene</p>	<p>Instrument and Conditions</p> <p>Model: Varian V-4300B Freq: 40 Mc/sec Scan Rates, cps/sec: STD (Spectrum A): 1.04 Other (Spectrum B): (Spectrum C): Temperature: 26°C</p>
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Proton Magnetic Resonance Spectral Data

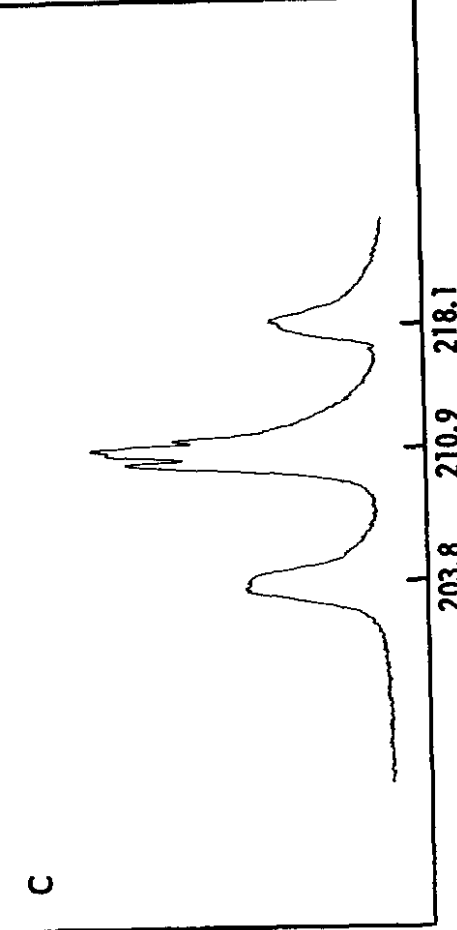
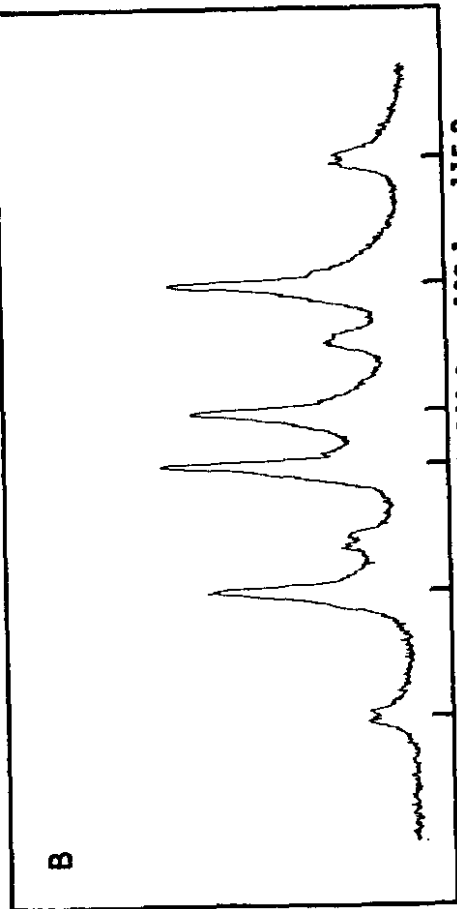
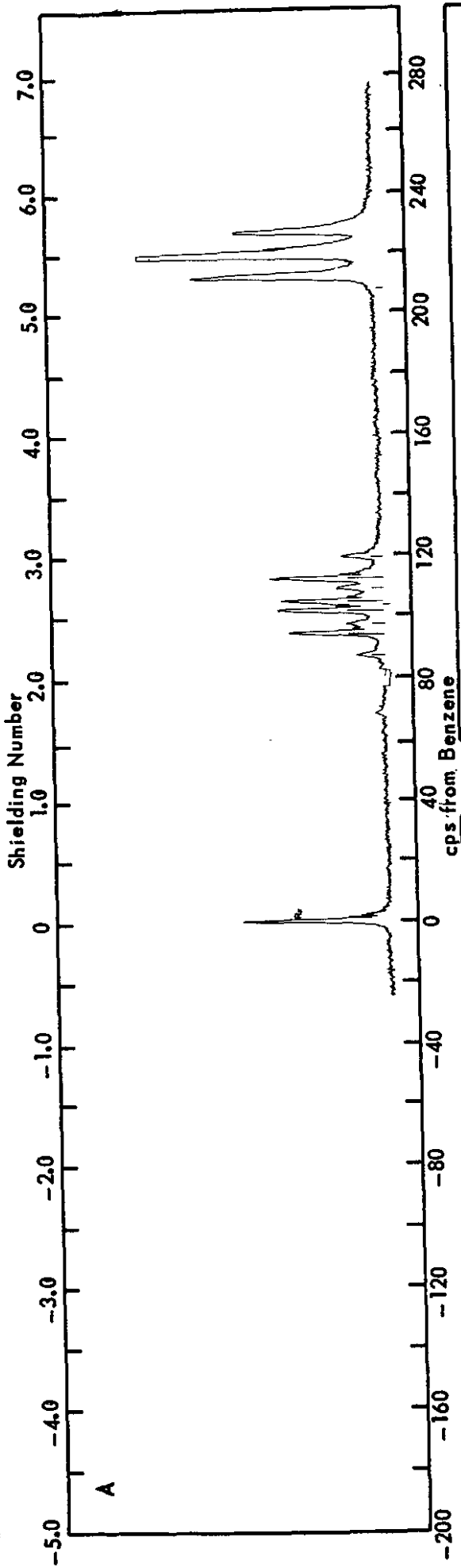


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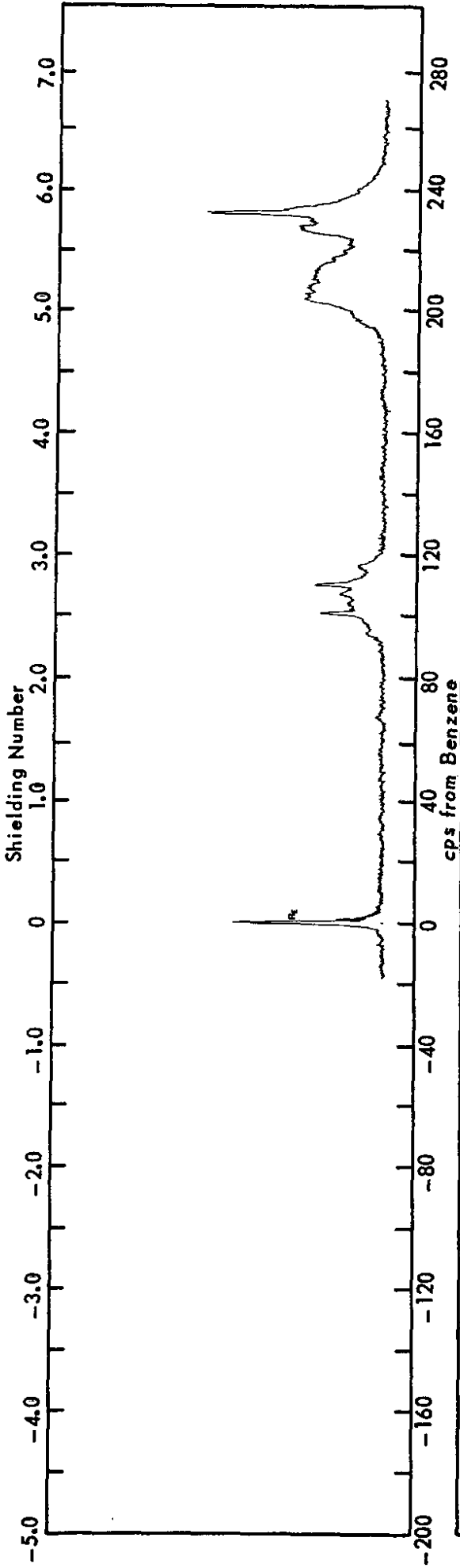
$\left(\begin{array}{c} \text{H} \\ \\ \text{C}_6\text{H}_4 \\ \\ \text{CH}_2\text{-O-P} \\ \\ \text{H} \end{array} \right)_3$	Sample Solvent	Ext. Reference Benzene	Instrument and Conditions Model: Varian V-4300B Freq.: 40 Mc/sec Scan Rates, cps/sec: STD (Spectrum A): 1.02 Other (Spectrum B): (Spectrum C): Temperature: 26°C
	Conc.	Int. Reference Benzene, 5% v	
Source and Purity Synthesis, crystallized		Resonance Position -29.0 cps from Benzene	

Proton Magnetic Resonance Spectral Data

Triethyl phosphorothioate

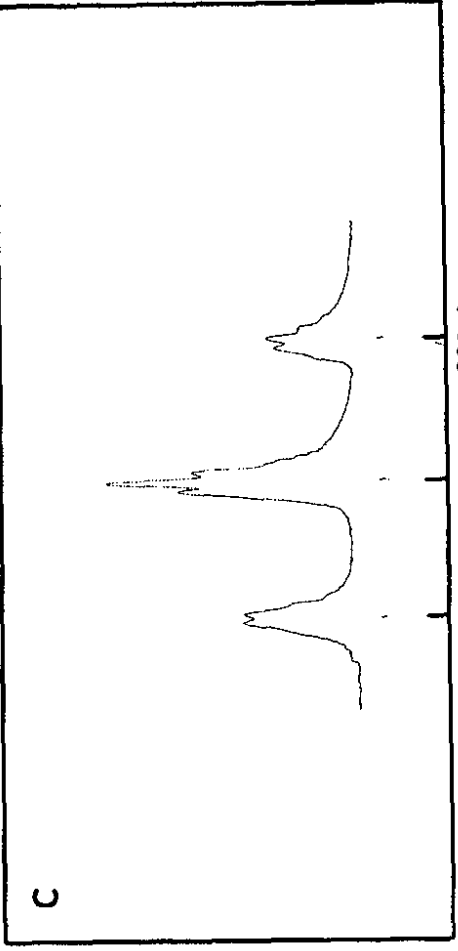
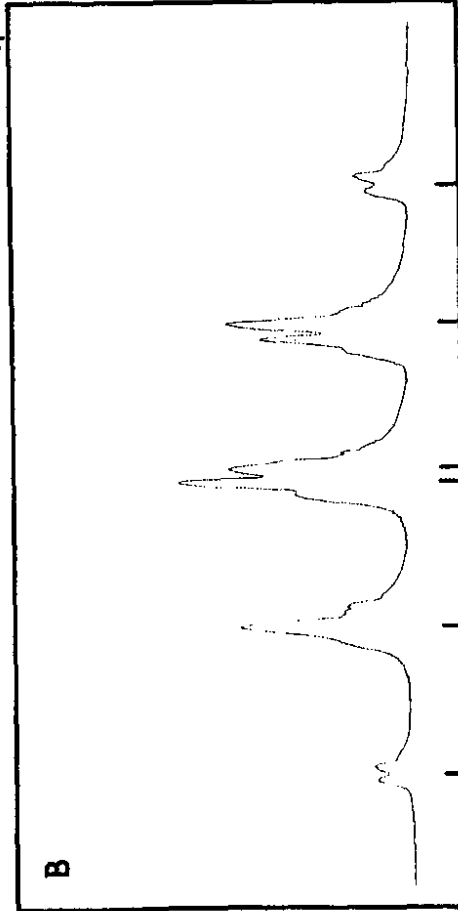
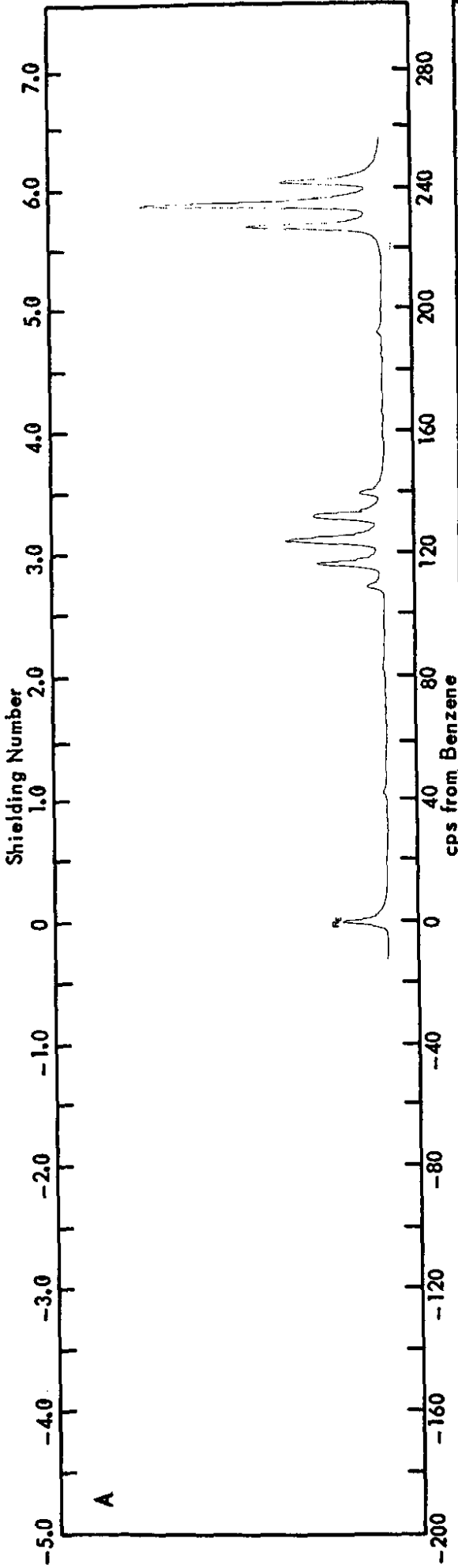


Sample		Ext. Reference	
		Benzene	
(CH ₃ -CH ₂ -O) ₃ PS		Int. Reference	
		Benzene 5% v	
Source and Purity Aldrich Chemical Co.		Resonance Position	
		-27.2 cps from Benzene	
Instrument and Conditions		Temperature: 26°C	
Model: Varian V-4300B		Scan Rates, cps/sec: STD (Spectrum A): 1.00 Other (Spectrum B): 0.29 (Spectrum C): 0.29	
Freq: 40 Mc/sec			

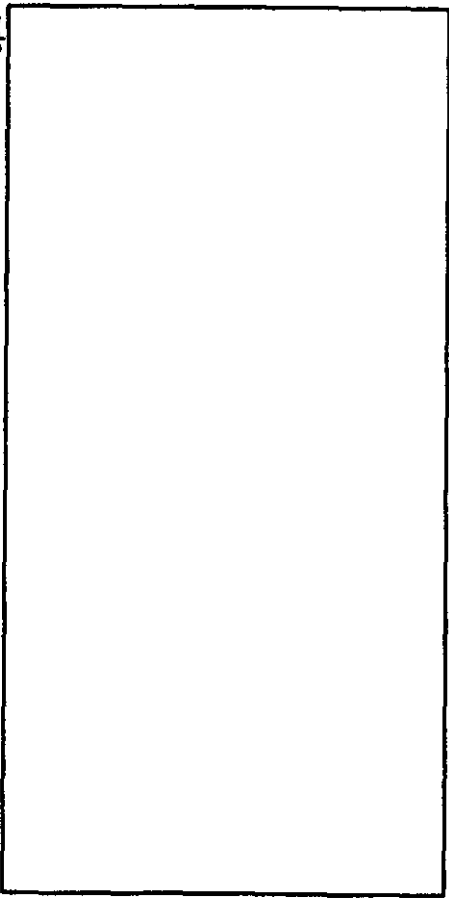
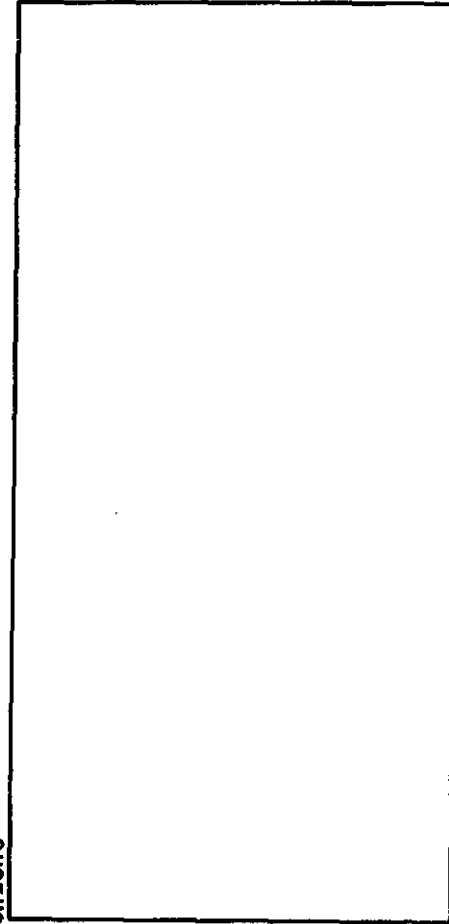
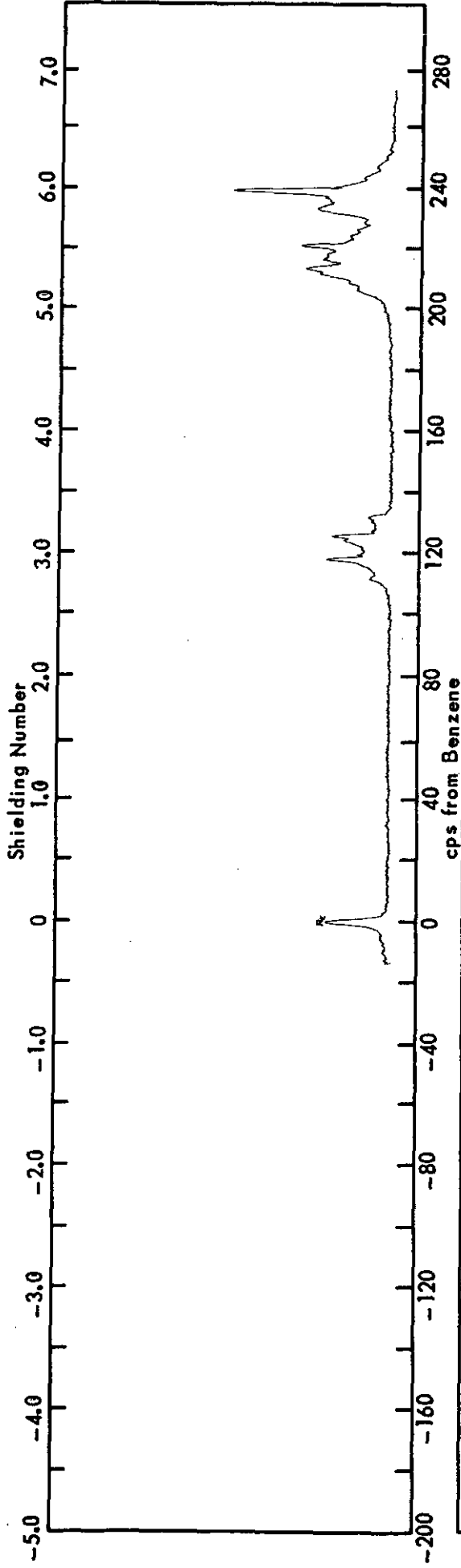


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<p>Sample</p> <p>(CH₃-CH₂-CH₂-CH₂-O)₃PS</p> <p>Source and Purity Virginia-Carolina Chemical Co.</p>	<p>Solvent</p> <p>Benzene, 5% v</p>	<p>Ext. Reference</p> <p>Benzene</p>	<p>Instrument and Conditions</p> <p>Model: Varian V-4300B Freq.: 40 Mc/sec Scan Rates, cps/sec: STD (Spectrum A): 1.05 Other (Spectrum B): (Spectrum C): Temperature: 26°C</p>
	<p>Conc.</p>		

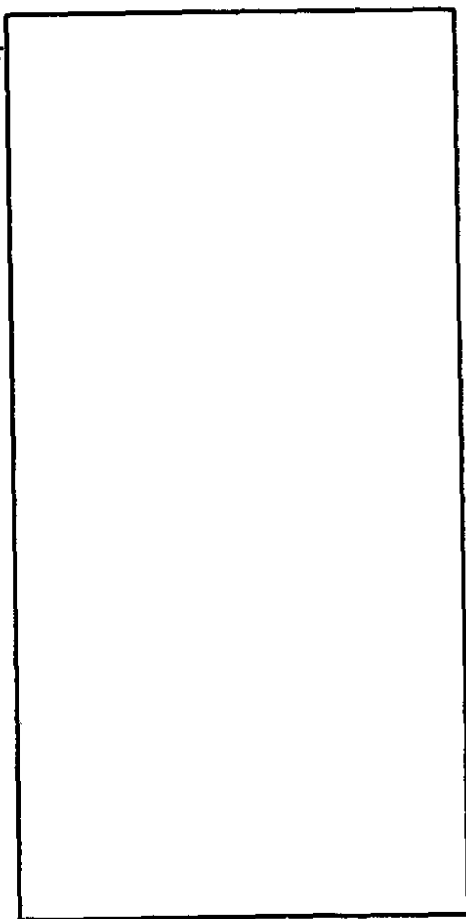
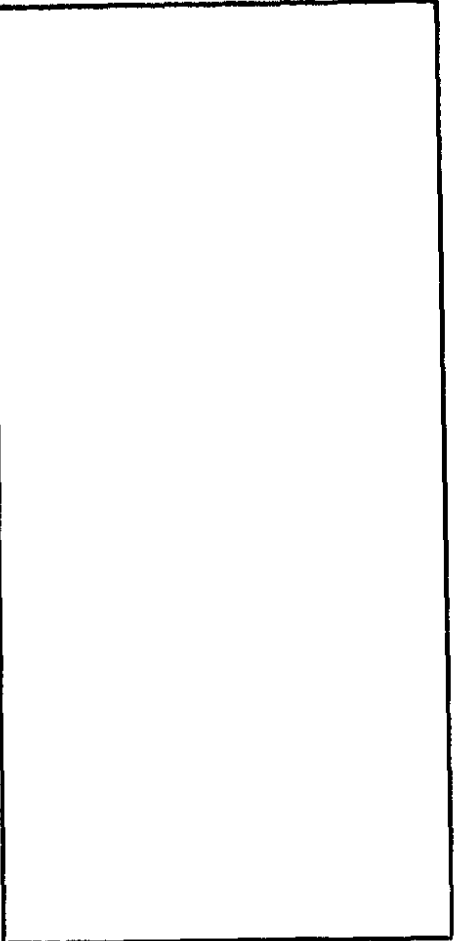
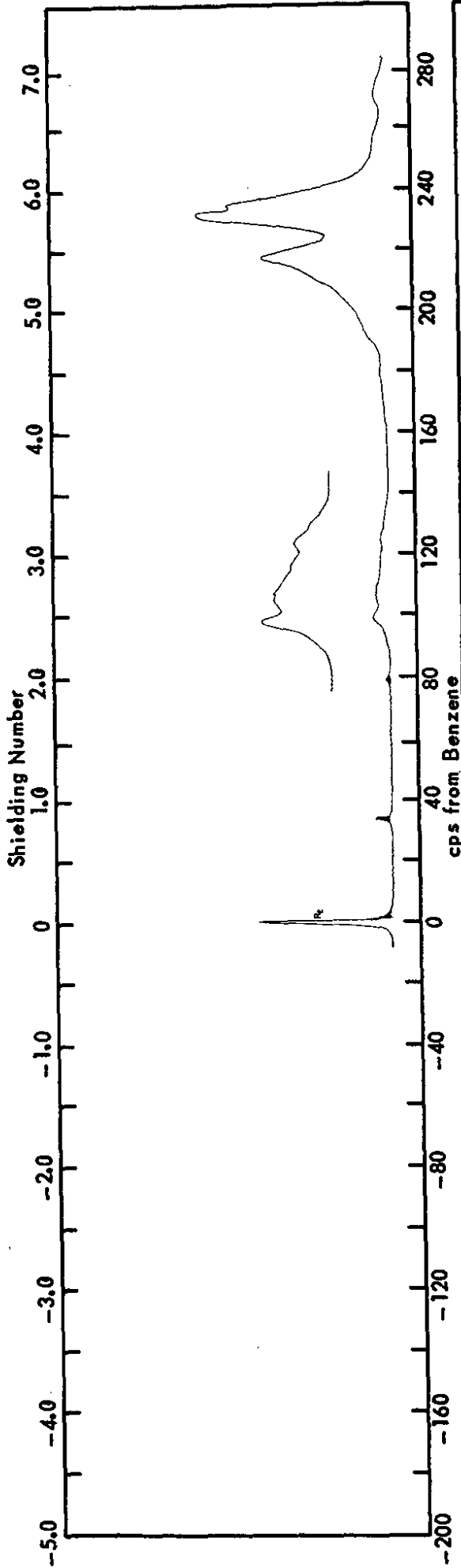


Sample		Ext. Reference		Instrument and Conditions	
		Benzene			
(CH ₃ -CH ₂ -O) ₃ P		Int. Reference		Model: Varian V-4300B Freq.: 40 Mc/sec Scan Rates, cps/sec: STD (Spectrum A): 1.03 Other (Spectrum B): 0.26 (Spectrum C): 0.23 Temperature: 26°C	
		Benzene, 5% v			
Source and Purity Eastman (Prac.); Aldrich Chemical Co.		Resonance Position -19.6cps from Benzene			

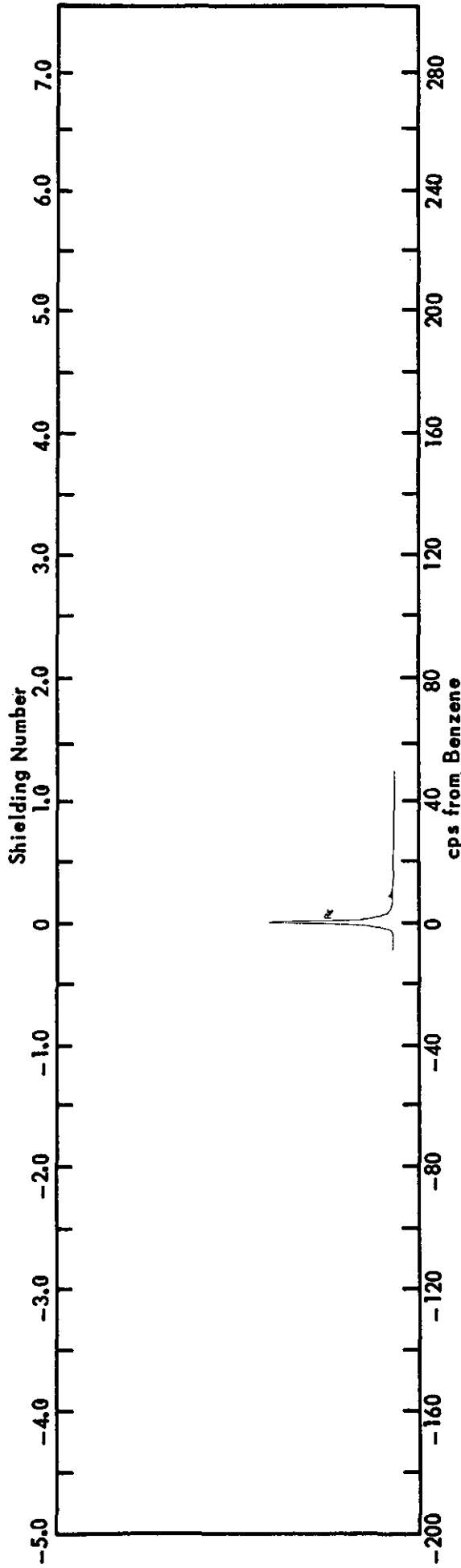


Sample		Ext. Reference		Instrument and Conditions	
(CH ₃ -CH ₂ -CH ₂ -CH ₂ -O) ₃ P	Solvent	Benzene		Model: Varian V-4300B	
	Conc.	Benzene, 5% v		Freq: 40 Mc/sec	
Source and Purity Eastman (Prac.)		Resonance Position		Scan Rates, cps/sec:	
		-18.4 cps from Benzene		STD (Spectrum A): 1.01	
				Other (Spectrum B): (Spectrum C):	
				Temperature: 26°C	

Proton Magnetic Resonance Spectral Data

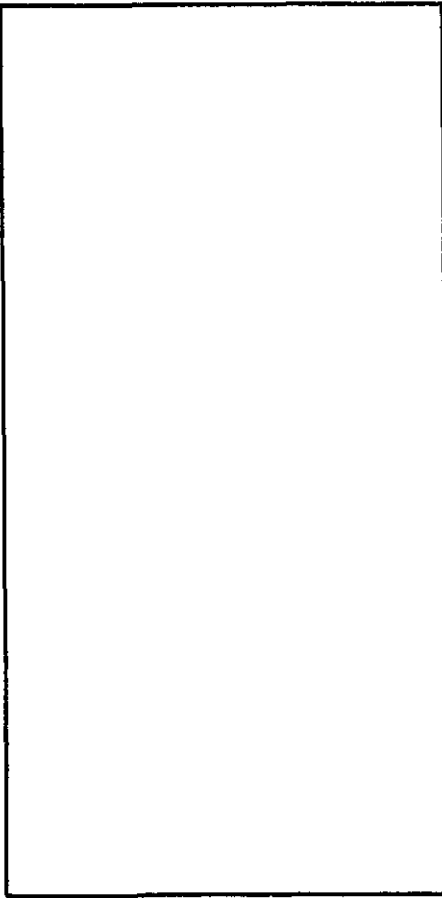
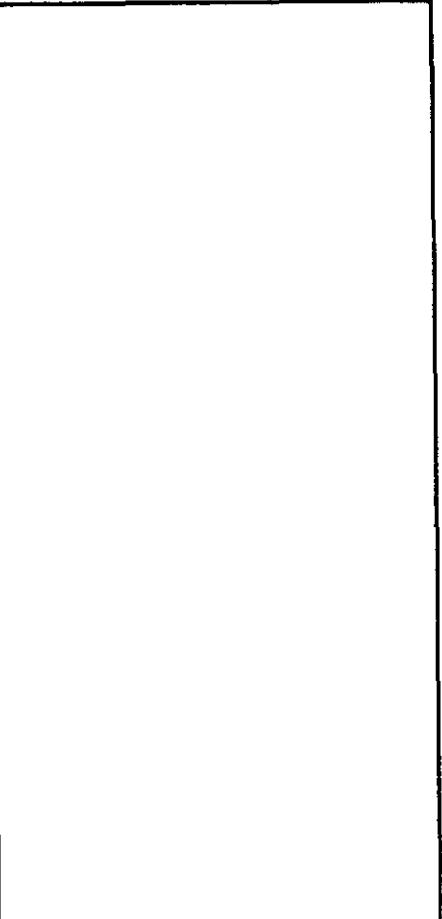
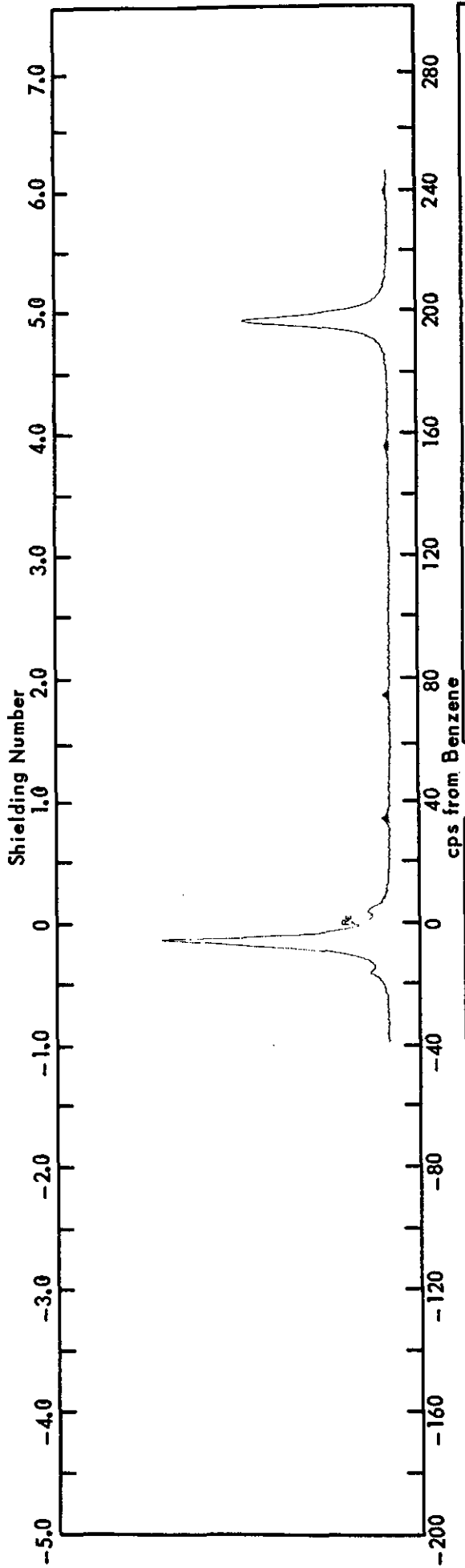


Sample		Instrument and Conditions	
		Model: Varian V-4300B Freq: 40 Mc/sec Scan Rates, cps/sec: STD (Spectrum A): 0.96 Other (Spectrum B): (Spectrum C): Temperature: 26°C	
$[CH_3-(CH_2)_8-CH_2-O]_3P$		Ext. Reference	Resonance Position cps from Benzene
		Benzene	
Source and Purity Shea Chemical Co.		Int. Reference	cps from Benzene
		Conc.	

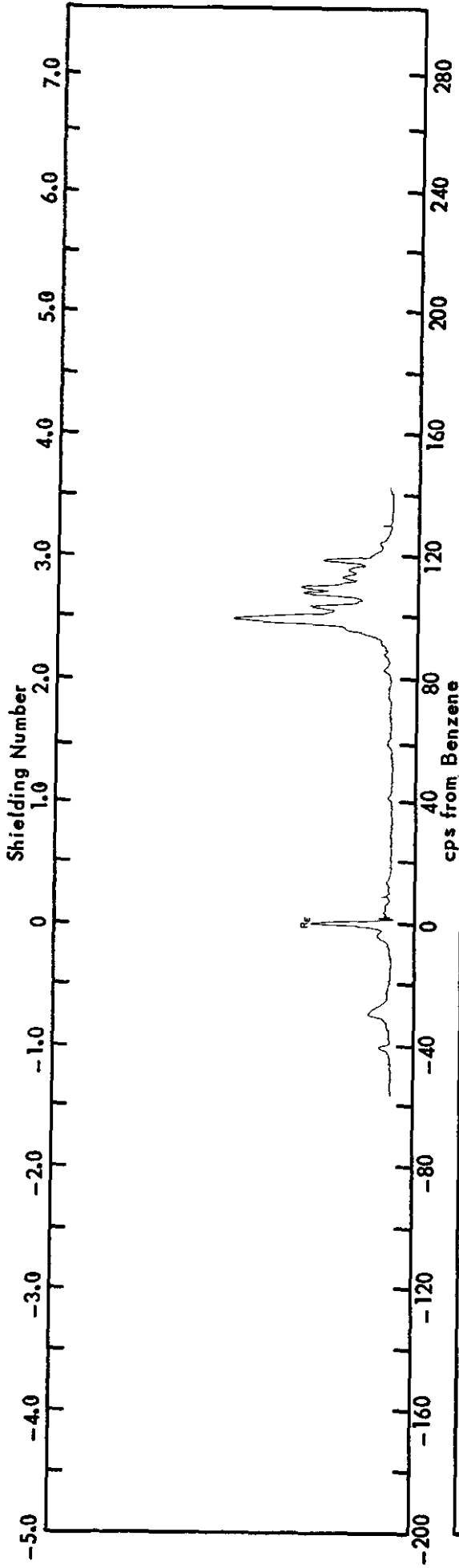


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<p style="text-align: center;">Sample</p> <div style="text-align: center;"> </div>	<p style="text-align: center;">Solvent</p>	<p style="text-align: center;">Ext. Reference</p> <p style="text-align: center;">Benzene</p>	<p style="text-align: center;">Instrument and Conditions</p> <p>Model: Varian V-4300B Freq.: 40 Mc/sec Scan Rates, cps/sec: STD (Spectrum A): 0.96 Other (Spectrum B): (Spectrum C): Temperature: 26°C</p>
	<p style="text-align: center;">Conc.</p>		
<p style="text-align: center;">Source and Purity</p> <p style="text-align: center;">Eastman (white label)</p>		<p style="text-align: center;">Resonance Position</p> <p style="text-align: center;">cps from Benzene</p>	



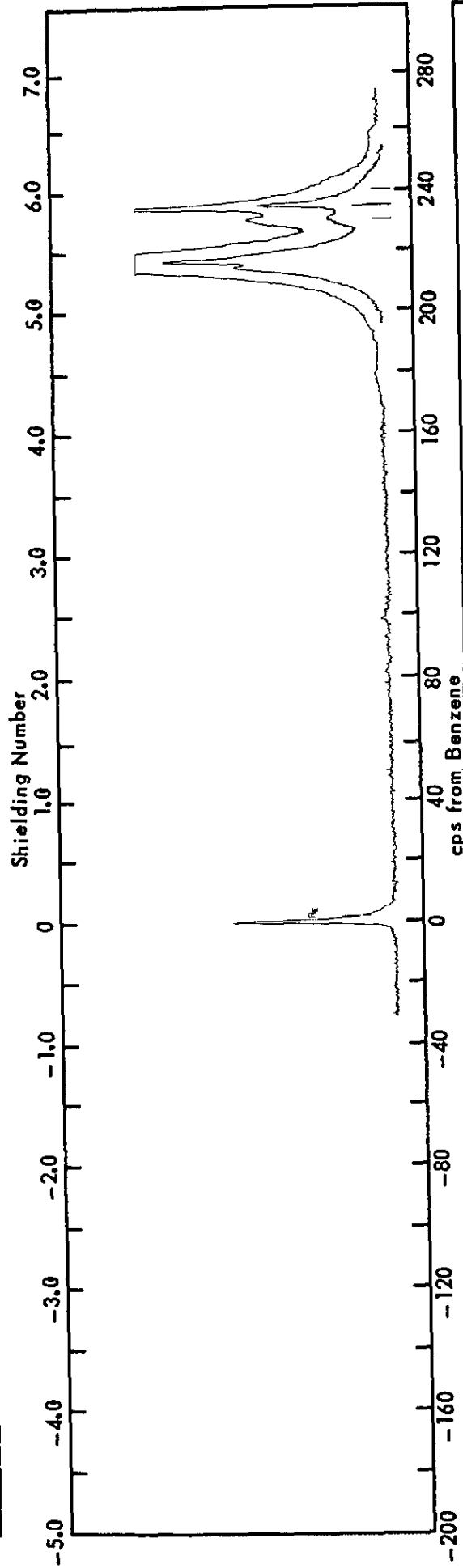
<p>Sample</p> <chem>Cc1ccc(Oc2ccc(C)cc2)cc1</chem> <p>(CH₃)₃P</p>	Solvent	<p>Ext. Reference Benzene</p> <p>Int. Reference</p> <p>Resonance Position cps from Benzene</p>	<p>Instrument and Conditions</p> <p>Model: Varian V-4300B</p> <p>Freq.: 40 Mc/sec</p> <p>Scan Rates, cps/sec: STD (Spectrum A): 1.06 Other (Spectrum B): (Spectrum C): Temperature: 26°C</p>
	Conc.		
	<p>Source and Purity</p> <p>Eastman (white label)</p>		



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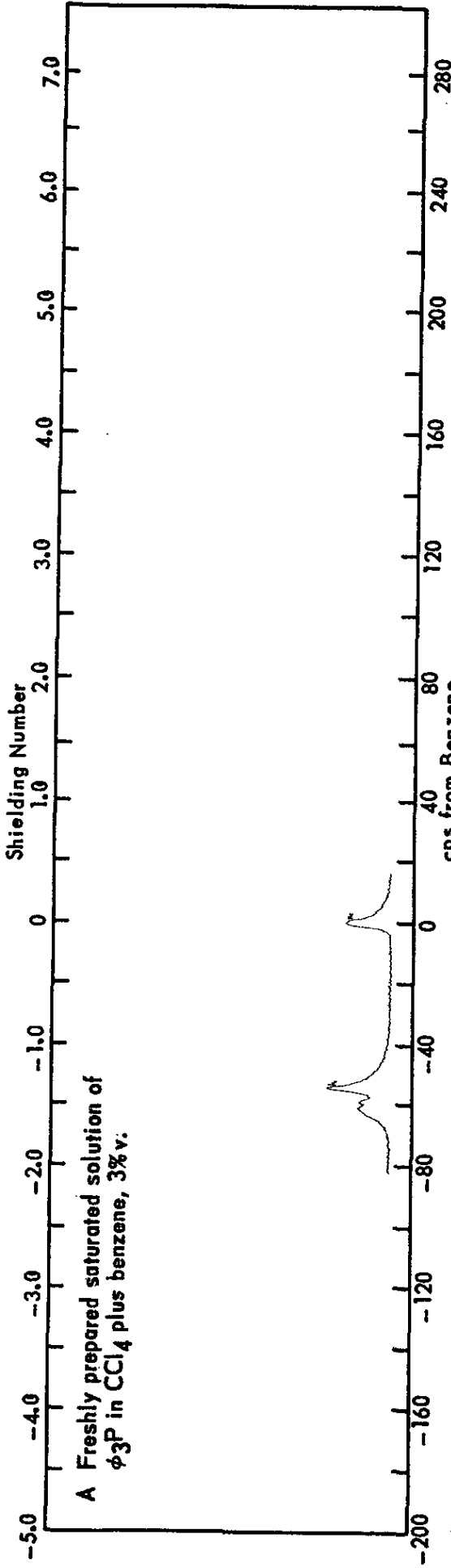
Sample		Instrument and Conditions	
$ \begin{array}{c} \text{CH}_2\text{-O} \quad \text{O-CH}_2 \\ \quad \quad \\ \text{P-O-CH}_2\text{-CH}_2\text{-O-P} \\ \quad \quad \\ \text{CH}_2\text{-O} \quad \text{O-CH}_2 \end{array} $	Solvent	Model: Varian V-4300B Freq: 40 Mc/sec Scan Rates, cps/sec: STD (Spectrum A): 1.07 Other (Spectrum B): (Spectrum C): Temperature: 26°C	
	Conc.		
Source and Purity Shea Chemical Co.		Ext. Reference Benzene	
		Int. Reference	
		Resonance Position cps from Benzene	



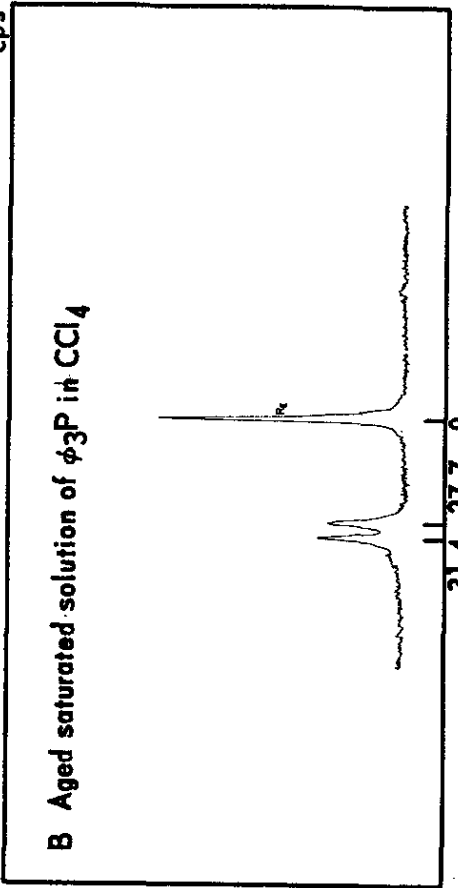
Sample		Ext. Reference	
(CH ₃ CH ₂ CH ₂ CH ₂) ₃ P		Benzene	
		Int. Reference	
Source and Purity		Resonance Position	
Shea Chemical Co.		cps from Benzene	

Instrument and Conditions	
Model: Varian V-4300B	
Freq.: 40 Mc/sec	
Scan Rates, cps/sec:	
STD (Spectrum A): 1.09	
Other (Spectrum B):	
(Spectrum C):	
Temperature: 26°C	

Sample		Ext. Reference	
(CH ₃ CH ₂ CH ₂ CH ₂) ₃ P		Benzene	
		Int. Reference	
Source and Purity		Resonance Position	
Shea Chemical Co.		cps from Benzene	

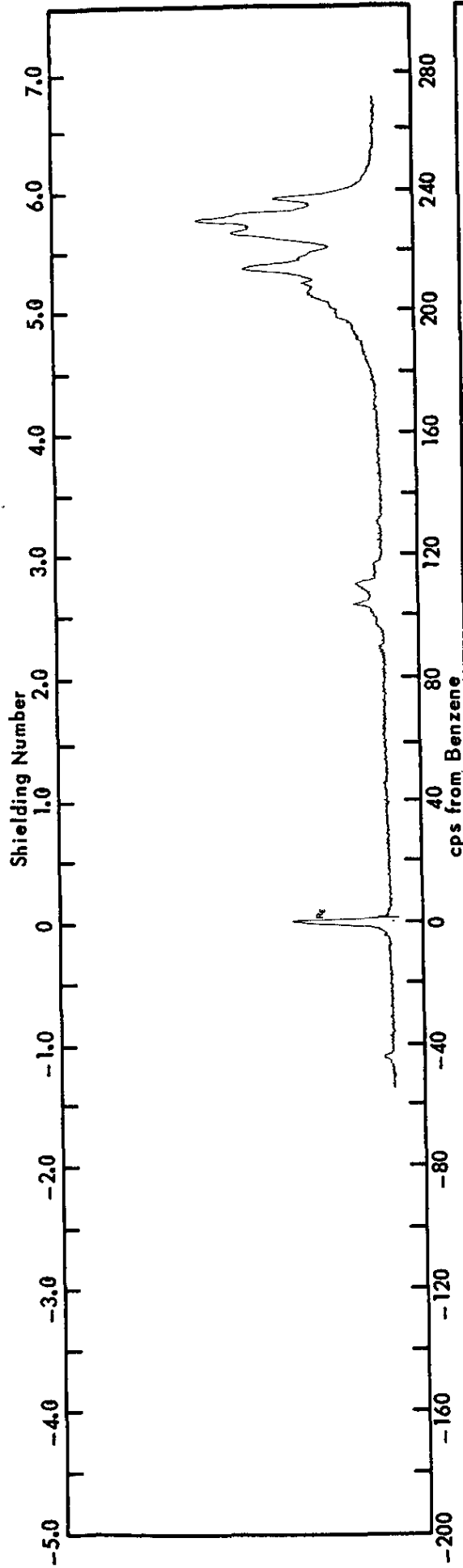


A Freshly prepared saturated solution of ϕ_3P in CCl_4 plus benzene, 3% v.



B Aged saturated solution of ϕ_3P in CCl_4

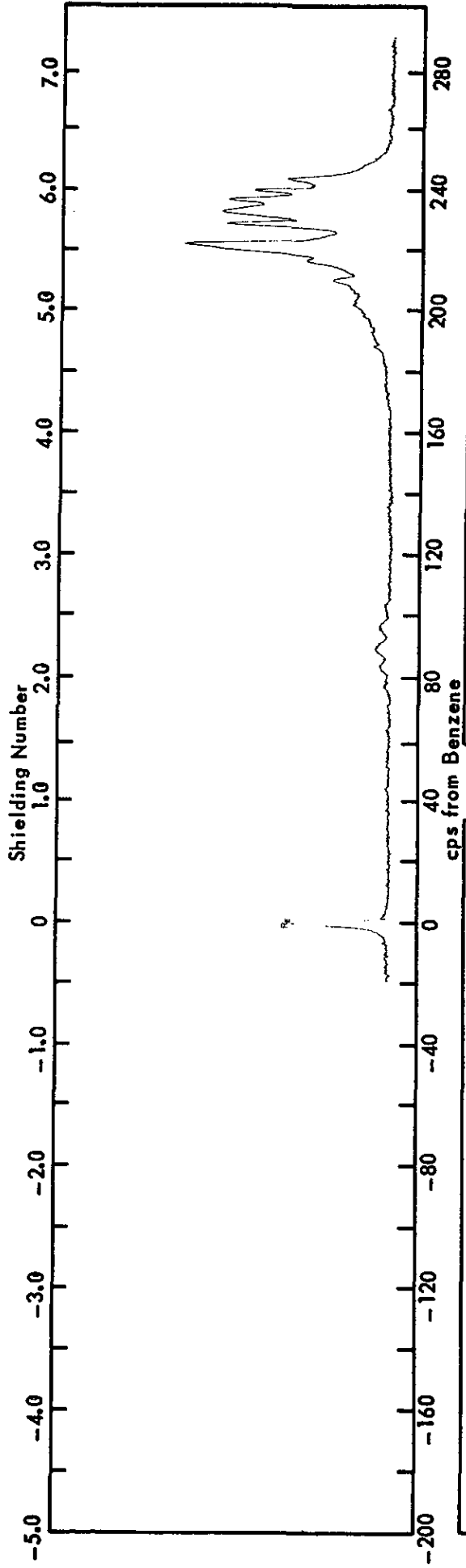
<chem>C1=CC=C(C=C1)P(C2=CC=CC=C2)C3=CC=CC=C3</chem> Source and Purity Eastman		Solvent		Ext. Reference	
		CCl_4 Conc. Saturated		Benzene	
				Int. Reference	
				Benzene, 3% v	
				Resonance Position	
				-24.3 cps from Benzene	
Instrument and Conditions					
Model: Varian V-4300B					
Freq.: 40 Mc/sec					
Scan Rates, cps/sec:					
STD (Spectrum A): 0.28					
Other (Spectrum B): 0.97					
(Spectrum C):					
Temperature: 26°C					



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Sample		Ext. Reference		Instrument and Conditions	
$\text{CH}_3 - (\text{CH}_2)_3 - \text{O} - \text{P}(\text{O})(\text{CH}_2 - \text{CH}(\text{CH}_3)_2)$	Solvent	Benzene		Model: Varian V-4300B	
	Conc.	Benzene, 5% v		Freq.: 40 Mc/sec	
Source and Purity Synthesis		Resonance Position		Scan Rates, cps/sec:	
		-28.4 cps from Benzene		STD (Spectrum A): 1.01	
				Other (Spectrum B):	
				(Spectrum C):	
				Temperature: 26°C	



Blank area for additional data or notes.

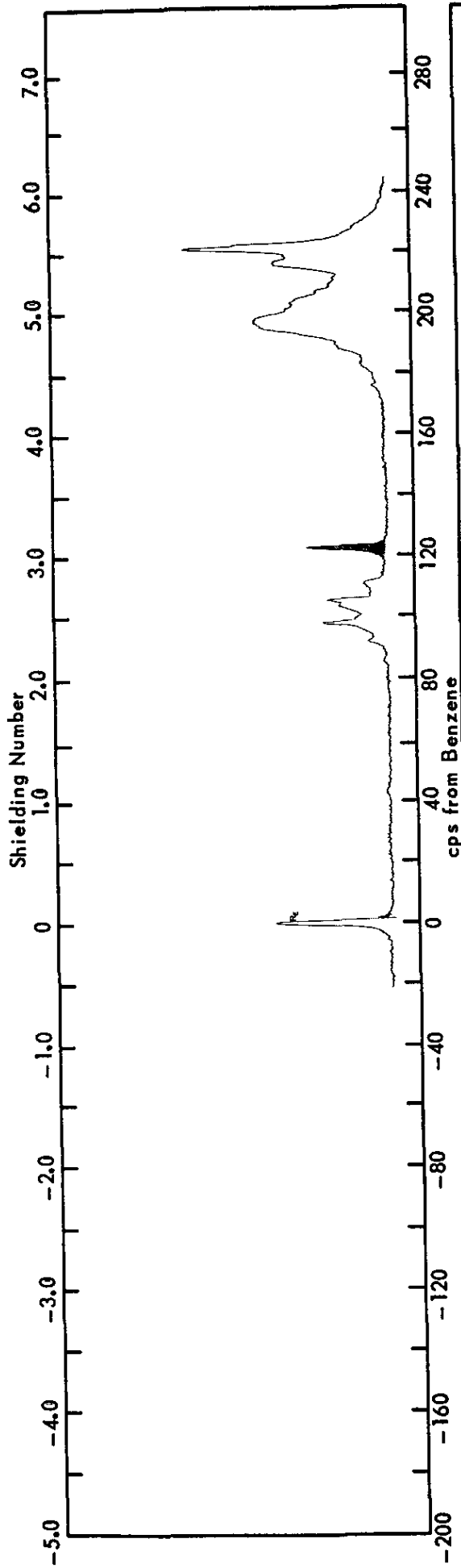
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Sample		Ext. Reference		Instrument and Conditions	
$\text{CH}_3\text{-CH}_2\text{-CH-O}$ CH_3 $(\text{CH}_3\text{-CH}_2\text{-CH-})\text{PO}$ $\text{CH}_3/2$	Solvent	Benzene		Model: Varian V-4300B	
	Conc.	Benzene, 5% v		Freq: 40 Mc/sec	
Source and Purity Synthesis		Resonance Position		Scan Rates, cps/sec:	
		-28.5 cps from Benzene		STD (Spectrum A): 1.02	
				Other (Spectrum B): (Spectrum C):	
				Temperature: 26°C	

Di-n-butyl n-butylphosphonate

Serial No. 41

Proton Magnetic Resonance Spectral Data

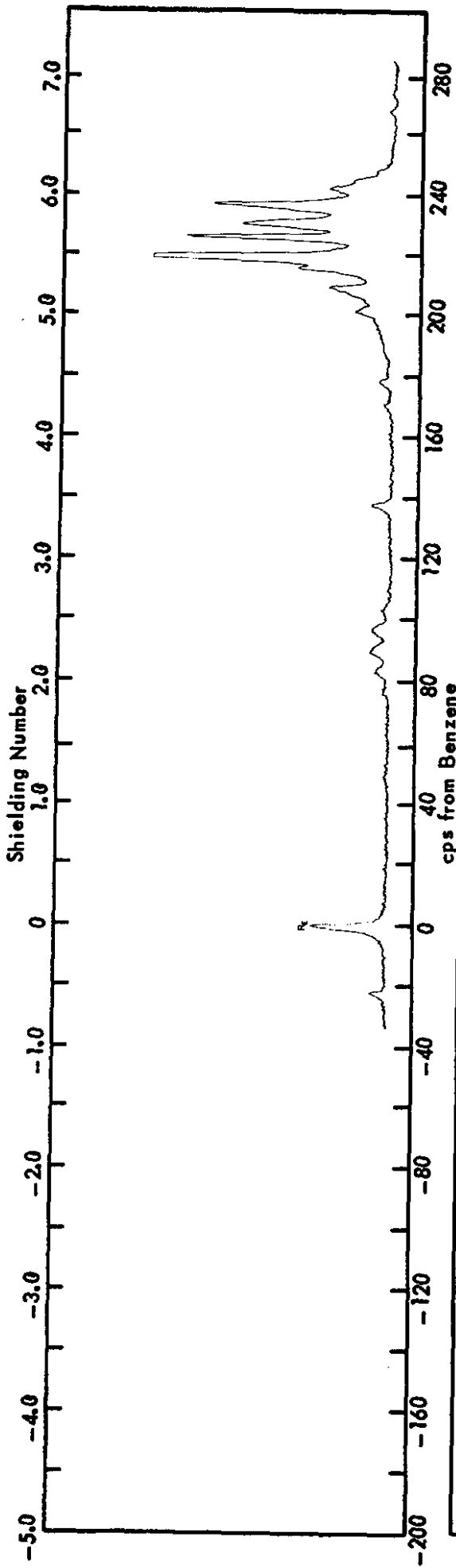


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Blank area for additional data or notes.

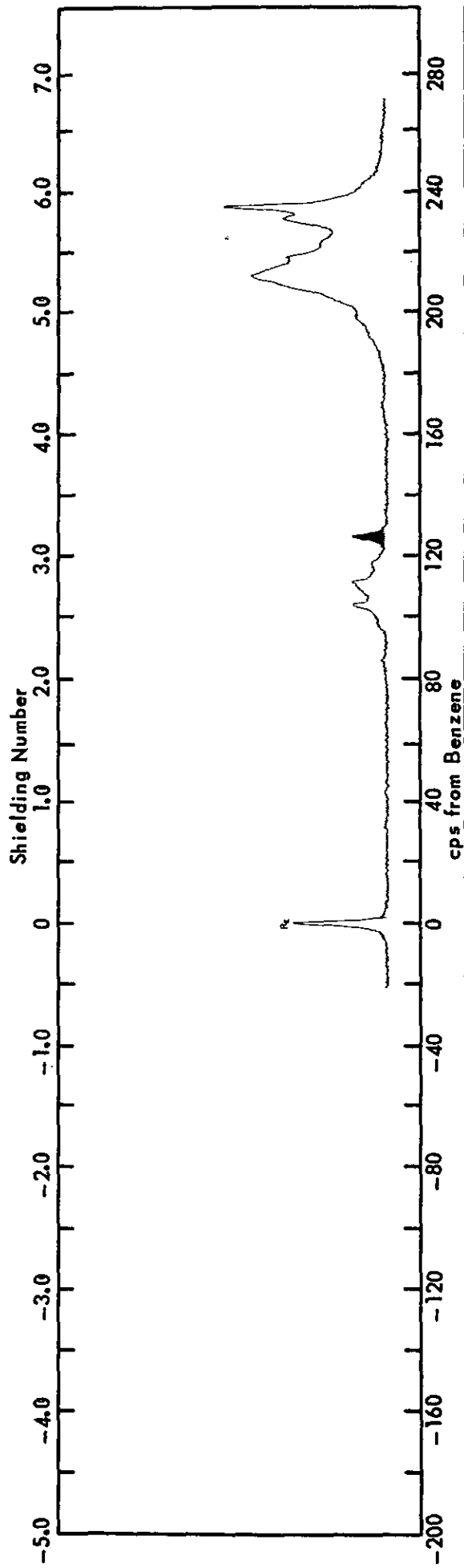
Sample		Ext. Reference		Instrument and Conditions	
$(CH_3-CH_2-CH_2-CH_2O)_2(CH_3-CH_2-CH_2-CH_2)PO$		Benzene		Model: Varian V-4300B	
		Benzene, 5% v		Freq: 40 Mc/sec	
Conc.		Resonance Position		Scan Rates, cps/sec: 1.01	
Source and Purity Virginia - Carolina Chemical Co.		-23.7 cps from Benzene		Other (Spectrum B):	
				(Spectrum C):	
				Temperature: 26°C	

Proton Magnetic Resonance Spectral Data



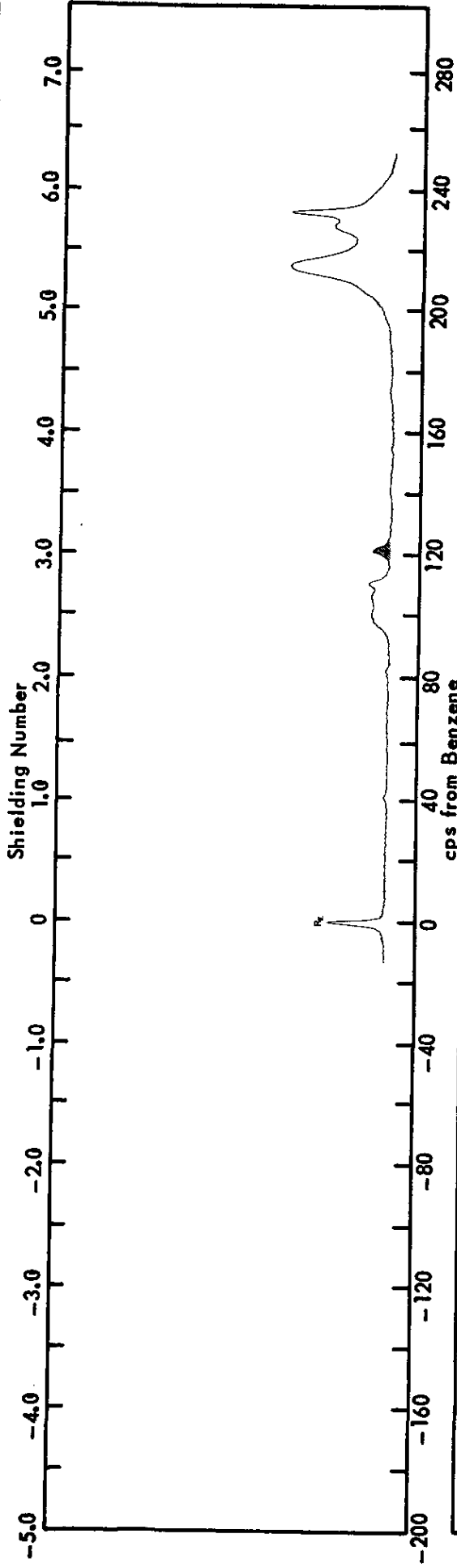
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<p>Sample</p> $\left(\begin{array}{c} \text{CH}_3\text{CH}_2\text{CH}-\text{O}- \\ \\ \text{CH}_3 \end{array} \right)_2 \left(\begin{array}{c} \text{CH}_3-\text{CH}-\text{CH}_2-\text{PO} \\ \\ \text{CH}_3 \end{array} \right)$	<p>Solvent</p>	<p>Ext. Reference Benzene</p>	<p>Instrument and Conditions</p> <p>Model: Varian V-4300B Freq.: 40 Mc/sec Scan Rates, cps/sec: STD (Spectrum A): 1.02 Other (Spectrum B): (Spectrum C): Temperature: 26°C</p>
	<p>Conc.</p>	<p>Int. Reference Benzene, 5% v</p>	
<p>Source and Purity Synthesis</p>	<p>Resonance Position -21.4 cps from Benzene</p>		



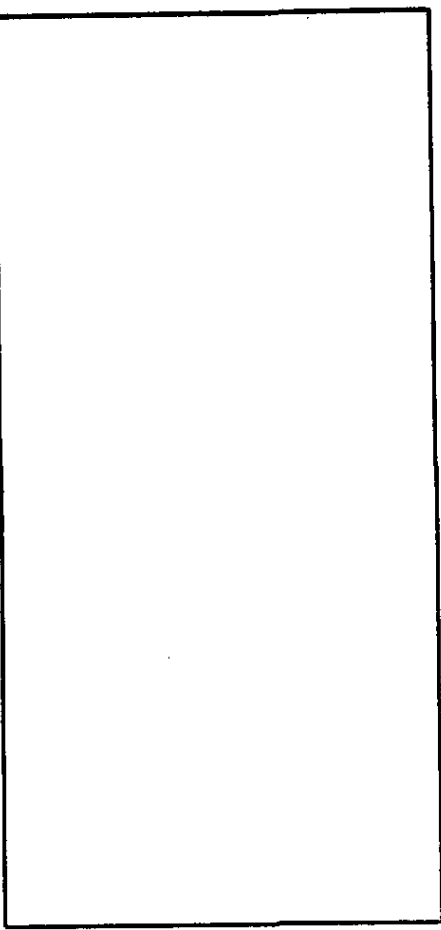
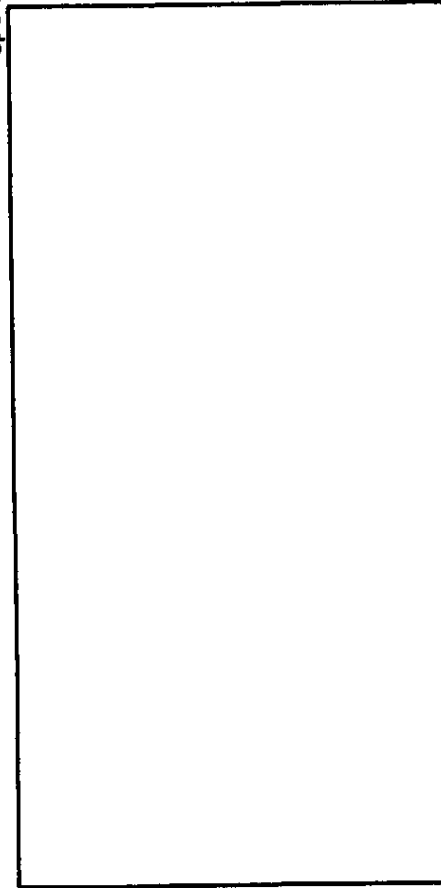
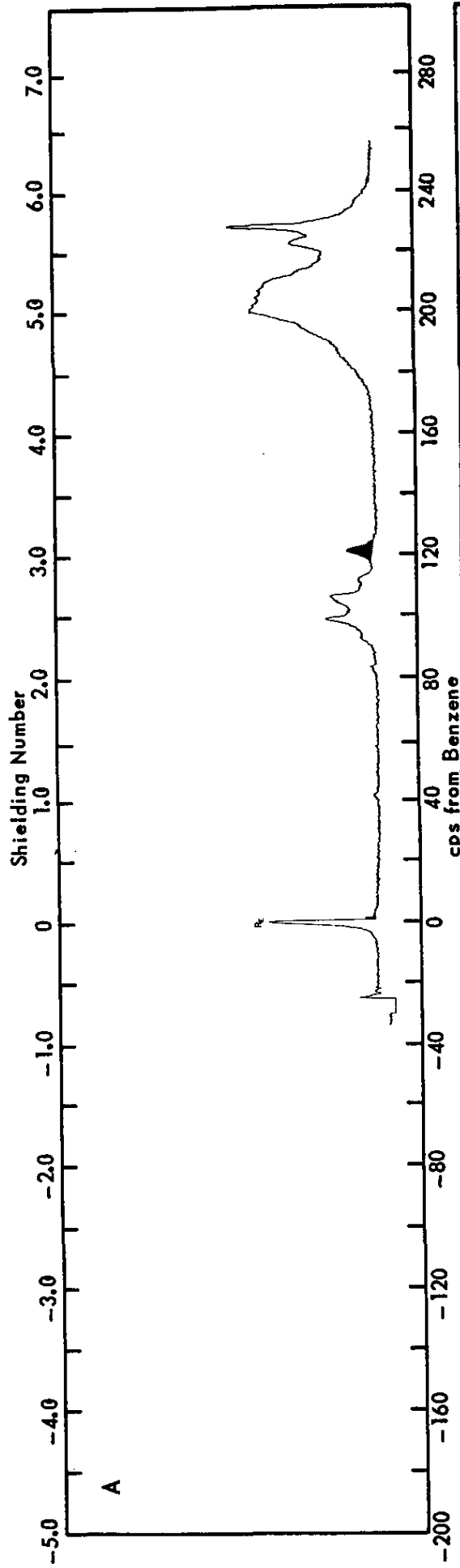
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Sample		Instrument and Conditions	
$\left[\text{CH}_3(\text{CH}_2)_4 \cdot \text{O} \right]_2 \left[\text{CH}_2(\text{CH}_2)_4 \cdot \text{PO} \right]$	Solvent	Model: Varian V-4300B Freq.: 40 Mc/sec Scan Rates, cps/sec: STD (Spectrum A): 1.01 Other (Spectrum B): (Spectrum C): Temperature: 26°C	
	Conc.		
Source and Purity Shea Chemical Co.			
		Ext. Reference	
		Benzene	
		Int. Reference	
		Benzene, 5% v	
		Resonance Position	
		-23.2 cps from Benzene	

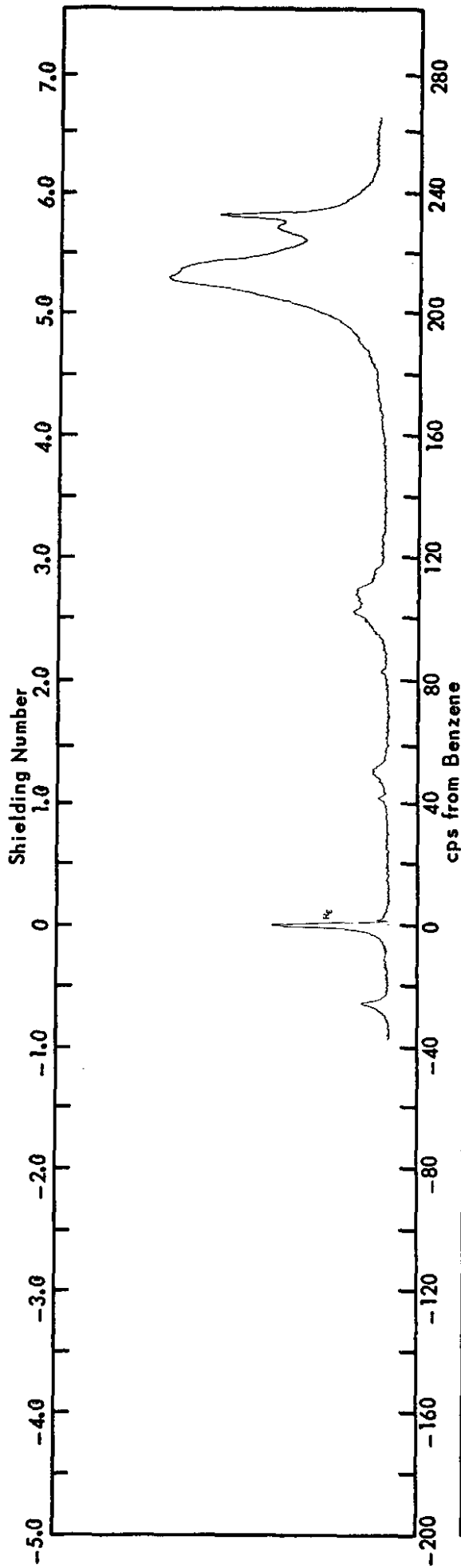


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Sample	Instrument and Conditions	
$(CH_3 \cdot CH_2 \cdot CH_2 \cdot CH_2 \cdot CH \cdot CH_2 \cdot O \cdot)_2 (ClCH_2 \cdot)PO$ C_2H_5 Source and Purity Monsanto Chemical Co.	Ext. Reference	Model: Varian V-4300B
	Benzene	Freq: 40 Mc/sec
	Int. Reference	Scan Rates, cps/sec:
Benzene, 5% v	Resonance Position	STD (Spectrum A): 1.03
-24.4 cps from Benzene	(Spectrum B):	Other (Spectrum C):
	Temperature: 26°C	



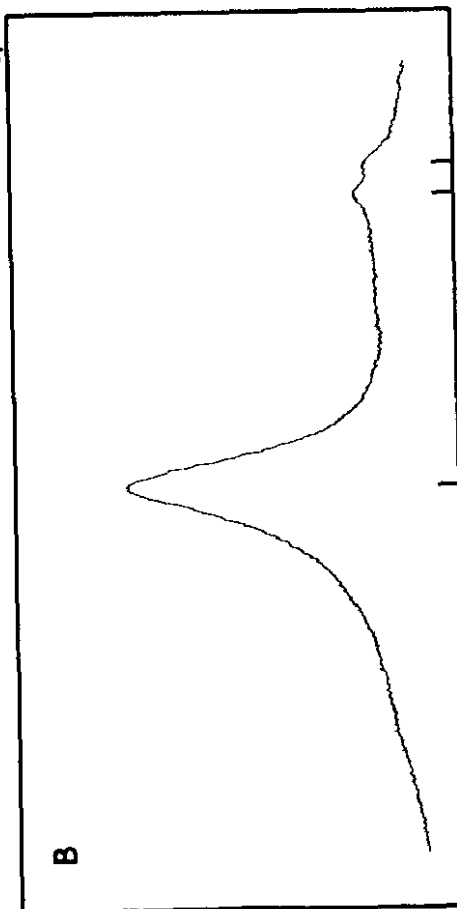
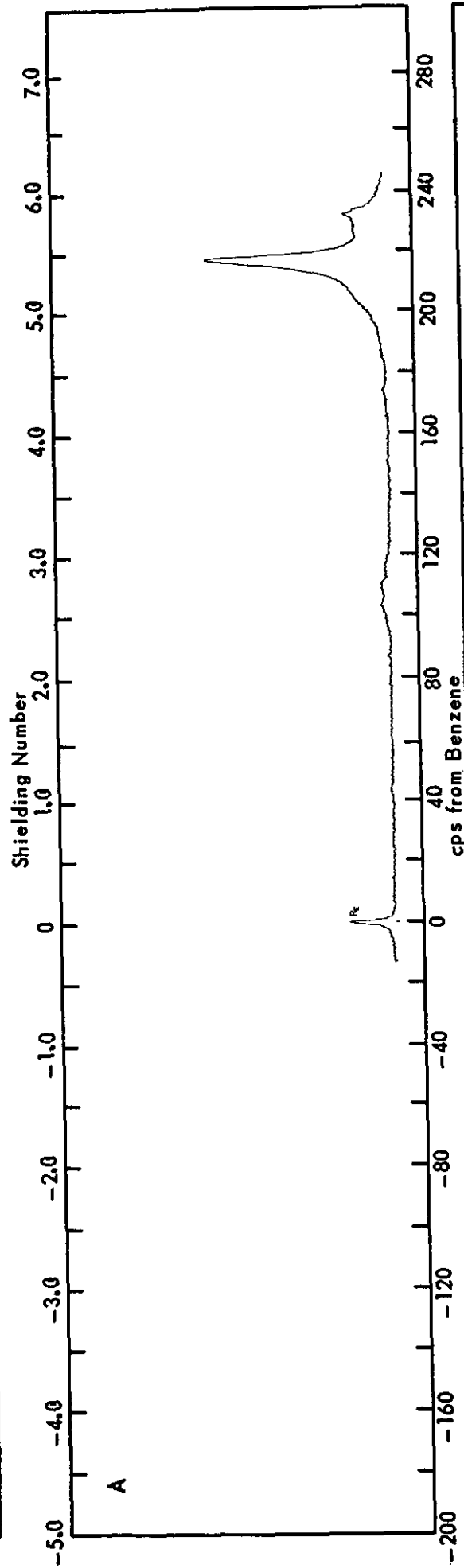
<p>Sample</p> $(CH_3CH_2CH_2CH_2-O)_2 \left(\begin{array}{c} CH_2-CH_2 \\ \quad \\ CH_2 \quad CH \\ \quad \\ CH_2-CH_2 \end{array} \right) \cdot PO$ <p>Source and Purity Shea Chemical Co.</p>	<p>Ext. Reference Benzene</p> <p>Int. Reference Benzene, 5% v</p> <p>Resonance Position -24.2 cps from Benzene</p>	<p>Instrument and Conditions</p> <p>Model: Varian V-4300B Freq.: 40 Mc/sec Scan Rates, cps/sec: STD (Spectrum A): 1.01 Other (Spectrum B): (Spectrum C): Temperature: 27°C</p>
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Sample		Ext. Reference		Instrument and Conditions	
$(C_6H_{13}-O-)_2(C_6H_{13}-)PO$	Solvent	Benzene		Model: Varian V-4300B	Temperature: 27°C
	Conc.	Benzene, 5%v		Freq.: 40 Mc/sec	
Source and Purity Synthesis	Resonance Position		Scan Rates, cps/sec:		
	-23.4 cps from Benzene		STD (Spectrum A): 1.04		
			Other (Spectrum B):		
			(Spectrum C):		

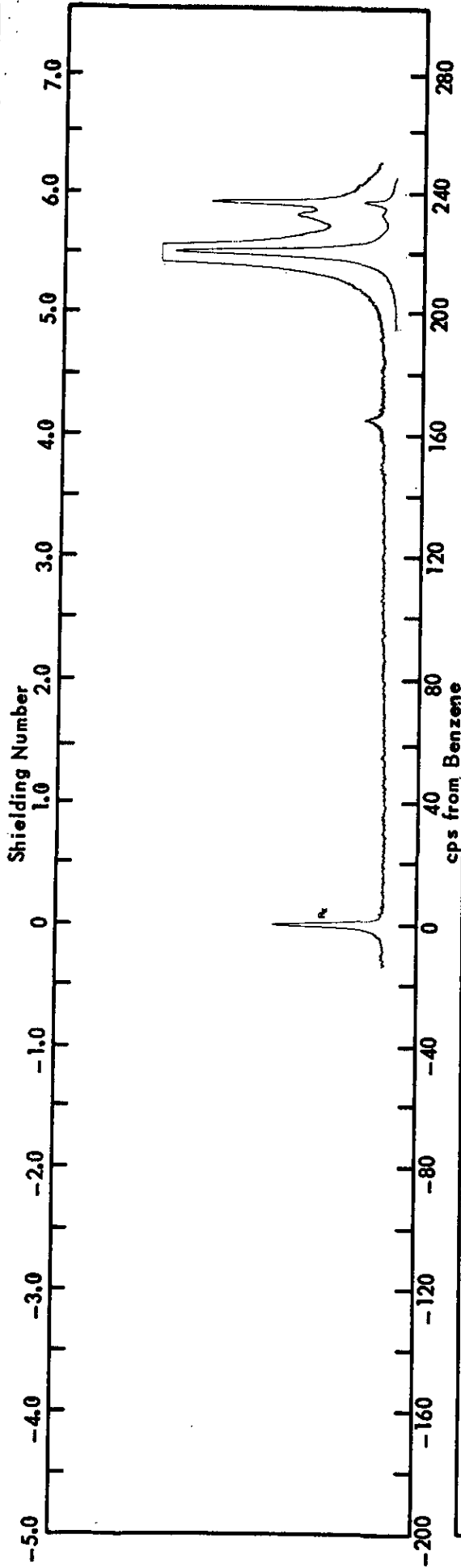


211.8 cps from Benzene

225.9 227.3

Sample		Ext. Reference		Instrument and Conditions	
		Benzene			
(C ₄ H ₉ O) ₂ (C ₁₄ H ₂₉)PO		Int. Reference		Model: Varian V-4300B	
		Benzene, 5% v		Freq.: 40 Mc/sec	
Source and Purity Synthesis		Resonance Position		Scan Rates, cps/sec:	
		-24.7 cps from Benzene		STD (Spectrum A): 1.01	
				Other (Spectrum B): 0.25	
				(Spectrum C):	
				Temperature: 27°C	

Proton Magnetic Resonance Spectral Data



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Sample		Ext. Reference		Instrument and Conditions	
(CH ₃ -CH ₂ -CH ₂ -CH ₂) ₃ PO		Benzene		Model: Varian V-4300B Freq.: 40 Mc/sec Scan Rates, cps/sec: STD (Spectrum A): 1.02 Other (Spectrum B): (Spectrum C): Temperature: 26°C	
		Benzene, 5% v			
Source and Purity Synthesis		Resonance Position -16.4 cps from Benzene			