The Mass Spectra of Some Silver Salts

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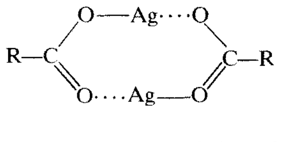
Fifteen of twenty-four silver(I) carboxylates examined give useful electron impact mass spectra. The compounds vaporize at moderate temperatures, apparently mainly as dimer with traces of higher oligomer in only a few cases. The molecular ion for the dimer is generally weak or absent, with the most abundant silver containing ion being $[Ag_2(O_2CR)]^+$ in most cases. Metastable defocusing and deuterium labeling experiments on silver acetate have established some of the fragmentation pathways. The reported loss of carbon dioxide from perfluorocarboxylates to give intense peaks for organosilver ions was not observed in this study. Attempts to obtain spectra on the silver salts of organic materials other than carboxylic acids were successful in several cases. Silver trifluoromethanesulfonate, although much less volatile, gives a spectrum and fragmentation very much like the carboxylates, whereas silver trifluoromethanethiolate gives a complex spectrum which suggests tetramer as a major gas phase species. Of three compounds examined which have silver to nitrogen bonding only silver(II) phthalocyanine is sufficiently volatile to give a spectrum without decomposition. The field desorption spectra of the four compounds examined all show the ions Ag_nX_{n-1} for X=acetate (n=1–6), X=p-chlorobenzoate (n=1–4), X=methanesulfonate (n=1–7) and X=p-toluenesulfonate (n=1–5).

There have been only a few reports on the electron impact (EI) mass spectra of organic silver compounds; a study of six silver(I) carboxylates[1] and three reports on isoelectronic compounds with carbon–silver bonds.[2] Species which are organic molecules cationized with silver have been observed in secondary ion mass spectrometry[3] and by field desorption from silver surfaces.[4] We have examined a wider range of silver carboxylates and some salts other than carboxylates. Field desorption (FD) spectra have been obtained on some of these salts. All of the compounds for which EI mass spectra were attempted are listed in Table 1.

Silver carboxylates

All of the silver containing ions and selected other ions for the silver carboxylates are listed in Table 2. The complete spectra have been submitted to the EPA/NIH mass spectral data base. As was observed by Adams *et al.*[1] the salts appear to vaporize almost entirely as the dimer.



A low abundance ion containing three silver atoms was observed in compound **3** by Adams *et al.* as the only evidence of higher oligomers. We observe ions with three atoms of silver in compounds **2**, **3**, **4**, **11** and **22**, and with four atoms of silver in compound **11**. These are of low abundance in all cases.

Table 1. Compounds for which EI mass spectra were attempted

Successful (silver containing ions observed)		Unsuccessful (silver containing ions absent)	
$AgO_2C—R$		$AgO_2C—R$	
$R=CH_3$	**(1)**	$R=(CH_2)_{12}CH_3$	**(8)**
$R=CH_2CH_3$	**(2)**	$R=(CH_2)_{16}CH_3$	**(9)**
$R=(CH_2)_2CH_3$	**(3)**		
$R=(CH_2)_4CH_3$	**(4)**		
$R=(CH_2)_6CH_3$	**(5)**		
$R=(CH_2)_8CH_3$	**(6)**		
$R=(CH_2)_{10}CH_3$	**(7)**		
$AgO_2C—R$			
$R=CF_3$	**(10)**		
$R=(CF_2)_2CF_3$	**(11)**		
$AgO_2C—R$		$AgO_2C—R$	
$R=CH_2CH_2C_6H_5$	**(12)**	$R=CH_2C{\equiv}N$	**(16)**
$R=C(CH_3)_3$	**(13)**	$R=CH_2CH{=}CH_2$	**(17)**
$R=C_3H_5$	**(14)**	$R=CH_2C_6H_5$	**(18)**
(cyclopropyl)		$R=CH{=}CH_2$	**(19)**
$R=C_{10}H_{15}$	**(15)**	$R=CH{=}C(CH_3)_2$	**(20)**
(1-adamantyl)			
$AgO_2C—C_6H_5$	**(21)**	$AgO_2C—C_6H_4—R$	
		$R=p\text{-}N(CH_3)_2$	**(23)**
$AgO_2C—C_6F_5$	**(22)**	$R=p\text{-}Cl$	**(24)**
		$R=o\text{-}OCH_3$	**(25)**
		$Ag^{II}(O_2C—C_5H_4N)_2$	**(26)**
		(picolinate)	
$AgO_3S—CF_3$	**(27)**	$AgO_3S—R$	
		$R=CH_3$	**(28)**
		$R=p\text{-tolyl}$	**(29)**
$AgSCF_3$	**(30)**	$AgSCH_2CH_3$	**(31)**
		$AgSCOCH_3$	**(32)**
		$AgOPO(OC_6H_5)_2$	**(33)**
$Ag^{II}(C_{32}H_{16}N_8)$	**(34)**	$Ag(C_6H_4N_3)$	**(35)**
(phthalocyanine)		(benzotriazole salt)	
		$Ag(C_5H_4N_5)$	**(36)**
		(adenine salt)	

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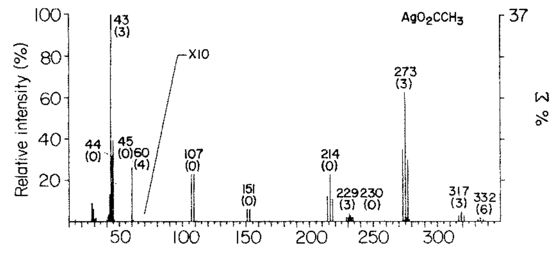


Figure 1. The 70 eV EI mass spectrum of silver acetate (**1**). The shift in mass for silver trideuteroacetate is given in parentheses.

The straight chain aliphatic carboxylates **1** through **7** all show very similar silver containing fragments which have been reported and discussed for compounds **1**, **2** and **3**.[1] The maximum relative intensity for most of the individual silver containing ions and the percentages of the total ion current carried in all of the silver containing ions (%Σ Ag in Table 2) is greatest for the propionate (**2**) then decreases with increasing chain length until in the two largest compounds, **8** and **9**, no silver containing ions are observed. In all of these compounds intense peaks are observed at the mass of the molecular ion for the corresponding carboxylic acid or at *m*/*z* 73, the expected $C_3H_5O_2$ fragment[5] of the carboxylic acid.

In order to establish the fragmentation pathways and to determine the origin of the carboxylic acid, we have carried out deuterium labeling and metastable defocusing experiments on silver acetate (**1**). The spectrum of **1** with the shift observed for the silver trideuteroacetate given in parentheses is shown in Fig. 1.

Scheme 1 shows a fragmentation scheme consistent with the labeling data and with the metastable ions observed by accelerating voltage scanning and by the direct analysis of daughter ions (DADI).[6] No metastable transition is found for the formation of acetic acid from *m*/*z* 332, but since the relative intensity of *m*/*z* 60 is not strongly dependent on the sample temperature and about 90% of the ion at *m*/*z* 60 is shifted four mass units in the deuterated compound it seems likely that the acetic acid is formed by EI induced fragmentation and not by thermal decomposition. One metastable transition observed by the DADI technique is not shown in Scheme 1, since we could not determine whether the transition was from *m*/*z* 273 to *m*/*z* 229 or to *m*/*z* 230. No metastable transition could

Table 2. Mass spectra of silver carboxylates AgO_2CR (including compositions of all silver containing ions and selected others)

	(1)		(2)		(3)		(4)		(5)		(6)		(7)		(10)	
	m/*z*	%[b]	*m*/*z*	%	*m*/*z*	%	*m*/*z*	%	*m*/*z*	%	*m*/*z*	%	*m*/*z*	%	*m*/*z*	%
Ag	107	4.6	107	61.5	107	13.7	107	4.0	107	3.0[c]	107	2.6	107	1.6[c]	107	135.6
AgC_2H_4[d]	—	—	135	8.0[c]	—	—	135	0.6	—	—	135	2.3	—	—	135	2.8
$AgCO_2$	151	1.2	151	11.1[c]	151	2.0	151	0.5	—	—	—	—	—	—	151	4.5
AgR	—	—	136	2.0[c]	—	—	—	—	—	—	—	—	—	—	—	—
AgR−H	—	—	135	8.0	149	2.0	177	0.4	—	—	233	0.6	—	—	NA[e]	—
AgO_2CR-H	—	—	—	—	—	—	221	1.5	—	—	277	0.6	305	0.4	NA	—
$AgNaO_2CR$	189	0.4	203	3.7	—	—	245	0.4	273	0.3	—	—	—	—	243	0.8
Ag_2	214	4.6	214	109.5	214	25.8	214	5.4	214	2.8	214	1.0	214	1.0	214	54.4
Ag_2H	—	—	215	18.7	215	13.9	215	3.8	215	2.8	215	1.2	215	0.3	NA	—
Ag_2O	230	0.7	230	9.6	—	—	230	0.2	—	—	—	—	—	—	230	11.0
Ag_2OH	—	—	231	3.3	—	—	231	0.2	—	—	—	—	—	—	NA	—
Ag_2F	NA	—	NA	—	NA	—	NA	—	NA	—	NA	—	NA	—	NA	—
Ag_2R	229	0.9	—	—	—	—	—	—	—	—	—	—	—	—	283[f]	0.8
Ag_2O_2CR	273	12.8	287	152.0	301	42.4	329	11.6	357	6.5	385	2.9	413	2.2	327	111.6
$Ag_2(O_2CR)CO_2$	317	1.1	331	4.7	345	0.4	—	—	—	—	—	—	—	—	—	—
$Ag_2(O_2CR)R$	—	—	—	—	344	0.4	400	0.3	—	—	—	—	—	—	—	—
$Ag_2(O_2CR)_2$	332	0.5	360	0.5	388	0.1	—	—	—	—	—	—	—	—	—	—
$Ag_3(O_2CR)_2$	—	—	467	0.6	495	0.1	551	0.4	—	—	—	—	—	—	—	—
Other apparent silver ions (Number of Ag atoms)	—	—	139 (1)	1.4	244 (2)	0.3	223 (2)	0.2[c]	249 (1)	1.0	—	—	—	—	123 (1)	3.3
	—	—	429 (3)	0.2	272 (2)	0.3	328 (2)	0.4[c]	356 (2)	0.4[c]	—	—	—	—	157 (1)	2.1
	—	—	—	—	387 (2)	0.1	—	—	—	—	—	—	—	—	—	—
	—	—	—	—	443 (3)	0.1	—	—	—	—	—	—	—	—	—	—
%Σ Ag[h]	—	10.3	—	43.5	—	17.9	—	4.1	—	2.4	—	1.2	—	0.4	—	62.5
RCO_2H	60	26.3	74	61.0	88	1.3	—	—	—	—	172	1.5	200	4.3	NA	—
RCO	43	100.0	57	100.0	71	64.7	99	100.0	127	65.6	155	54.7	183	54.8	97	19.7
R	15	1.0	29	41.3	43	38.3	71	26.4	99	1.9	—	—	—	—	69	100.0
$C_3H_5O_2$	NA	—	NA	—	73	36.0	73	41.4	73	70.2	73	100.0	73	100.0	NA	—
$CH_2{=}C(OH)_2$	NA	—	—	—	60	100.0	60	59.3	60	100.0	60	92.0	60	85.8	NA	—
CO_2H	45	39.4	45	39.2	45	14.8	45	10.1	45	10.2	45	9.3	45	13.4	NA	—
CO_2	44	32.1	44	25.0	44	16.2	44	5.9	44	7.2	44	8.1	44	10.5	44	38.7

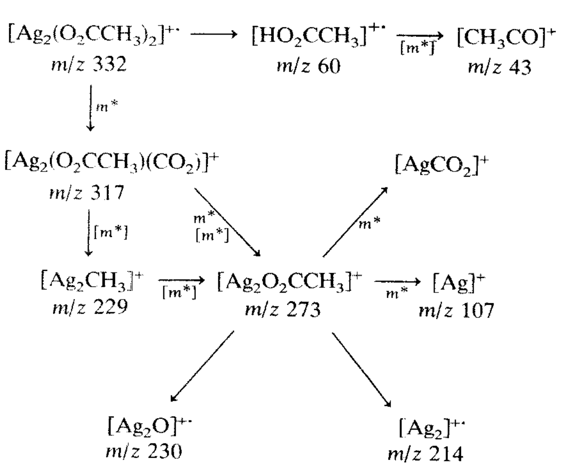
Table 2 (*continued*)

	(11)[a]		(12)[a]		(13)		(14)		(15)		(21)		(22)[a]	
	m/z	%	m/z	%	m/z	%	m/z	%	m/z	%	m/z	%	m/z	%
Ag	107	65.0	—	—	107	5.4	107	7.2	—	—	107	4.6	107	87.3
AgC_2H_4[d]	—	—	—	—	—	—	—	—	—	—	—	—	—	—
$AgCO_2$	151	2.0[c]	—	—	—	—	151	1.3	—	—	—	—	151	2.8[c]
AgR	—	—	—	—	—	—	148	0.4	—	—	—	—	274	3.8
AgR−H	NA	—	211	0.1	—	—	147	1.2	—	—	—	—	NA	—
AgO_2CR-H	NA	—	—	—	—	—	—	—	—	—	—	—	NA	—
$AgNaO_2CR$	343	0.8	279	0.2	—	—	—	—	—	—	—	—	—	—
Ag_2	214	20.1	214	0.2	214	8.1	214	7.5	—	—	214	2.8	214	5.7
Ag_2H	NA	—	215	0.3	—	—	215	2.1	—	—	—	—	NA	—
Ag_2O	230	3.3	—	—	—	—	230	0.6	—	—	—	—	—	—
Ag_2OH	NA	—	—	—	—	—	231	1.1	—	—	—	—	NA	—
Ag_2F	233	9.6	NA	—	NA	—	NA	—	NA	—	NA	—	233	8.7
Ag_2R	—	—	—	—	—	—	255	0.7	—	—	291	2.7	381	15.8
Ag_2O_2CR	427	39.2	363	1.4	315	16.9	299	17.6	393	0.7	335	4.3	425	2.5
$Ag_2(O_2CR)CO_2$	—	—	—	—	—	—	—	—	—	—	—	—	—	—
$Ag_2(O_2CR)R$	—	—	—	—	373	0.3	343	0.6	—	—	412	1.5	—	—
$Ag_2(O_2CR)_2$	—	—	—	—	—	—	384	0.4	—	—	456	<0.1	—	—
$Ag_3(O_2CR)_2$	747[g]	<0.1	—	—	—	—	—	—	—	—	—	—	—	—
Other apparent silver ions (Number of Ags)	—	—	257 (1)	0.2	372 (2)	0.5	—	—	421 (1)	0.1	368 (2)	<0.1	321 (3)	1.9
%Σ Ag[h]	—	33.9	—	0.6	—	12.5	—	12.1	—	0.2	—	4.1	—	19.1
RCO_2H	NA	—	150	31.0	—	—	86	0.6[c]	180	0.7	122	69.5	NA	—
RCO	—	—	133	3.1	85	2.7	69	100.0	163	0.8	105	100.0	—	—
R	169	7.6	105	30.4	57	100.0	41	39.2	135	100.0	77	65.5	167	5.9
$C_3H_5O_2$	NA	—	NA	—	NA	—	NA	—	NA	—	NA	—	NA	—
$CH_2{=}C(OH)_2$	NA	—	NA	—	NA	—	NA	—	NA	—	NA	—	NA	—
CO_2H	NA	—	45	3.1	45	5.8	45	8.1	—	—	45	3.1	NA	—
CO_2	44	32.0	44	7.1	44	11.1	44	10.0	44	10.5	44	4.1	44	76.3

[a] Base peaks are: **11**, m/z 100, C_2F_4; **12**, m/z 91, C_7H_7; **22**, m/z 334, $C_{12}F_{10}$.
[b] Intensity as percent of base peak. The intensity of silver containing peaks is the sum of all silver isotopes.
[c] Intensity corrected for overlapping of other ions.
[d] For compound **2**, measured mass is 134.9369; Calc. for AgC_2H_4, 134.9364.
[e] NA indicates that an ion of the indicated structure is not possible for this compound.
[f] Observed isotope pattern, m/z 283(0.25%)/285(0.39%)/287(0.16%), is not consistent with the presence of two silver atoms. See text.
[g] Observed only on Varian MAT CH-5 DF instrument.
[h] Percent of total ion current carried by all silver containing ions.

be found for either the formation or decay of the ion $[Ag_2]^{+\cdot}$ at *m/z* 214.

The two perfluorinated aliphatic salts (**10** and **11**) give silver containing ions very much like the unfluorinated compounds. The differences which do occur in the spectra are easily attributed to the expected effects of the presence of fluorine. Thus, the ions which require hydrogens such as $[RCO_2H]^{+\cdot}$ and $[CO_2H]^+$ are absent and $[Ag_2H]^+$ is replaced by $[Ag_2F]^+$, the percentage of the ion current carried by nonsilver containing ions decreases, and ions appear (such as the observed base peaks) which are typical of perfluorinated alkyl groups.

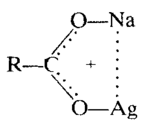


Scheme 1. Fragmentation pathways of silver acetate (**1**). Observed metastable transitions are labeled: m^* denotes observation using the DADI technique and $[m^*]$ observation by accelerating voltage scan.

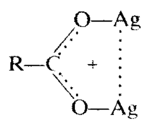
The data reported in the literature[1] for compounds **10** and **11** include substantial peaks for the ions $[Ag_2R]^+$ and $[Ag_2(O_2CR)R]^+$. As may be seen in Table 2, three of these peaks are absent in our spectra and the other is very weak and with an isotope pattern which does not correspond to that expected for two silver atoms. In an effort to resolve this discrepancy, spectra of compounds **10** and **11** were obtained using a Varian MAT CH-5 DF instrument. Ions are present at all four expected masses, but the relative intensities of these ions increase with sample concentration and the observed isotope ratios are not the 53/100/47 expected for two silver atoms. Instead the ratios are much closer to the 72/100/30 expected for one silver atom plus one copper atom. It thus appears that on our Varian MAT CH-5 DF instrument, for these two compounds only, the ions given as $[Ag_2R]^+$ and $[Ag_2(O_2CR)R]^{+\cdot}$ by Adams *et al.* are instead $[AgCu(O_2CR)]^+$ and $[AgCu(O_2CR)_2]^{+\cdot}$ formed by replacement of silver by copper within the instrument. Compound **11** when examined on an Hitachi-Perkin-Elmer RMU-6E also shows an ion at m/z 383 with an isotope pattern consistent with $[AgCu(O_2CR)]^+$ but not with $[Ag_2R]^+$. The copper block of the sample heater is an obvious source of copper in the Hitachi instrument, but the source is much less obvious in the Varian MAT instrument where the only visible copper is well outside the ionization chamber.

The remaining fourteen carboxylates listed in Table 1 were chosen to test the effects of variation in the structure of the acid. The most obvious effect is the pronounced tendency to decompose thermally if an additional multiple bond of any type is present. All except two of the unsuccessful compounds (**16**–**20** and **23**–**26**) are of this kind. In all cases one of the major products of thermal decomposition appears to be the corresponding carboxylic acid. Of the successful compounds with multiple bonds, silver benzoate (**21**) gives a spectrum in which the relative intensities of the silver containing ions decrease with increasing sample temperatures indicating that decomposition and volatilization begin at about the same temperature and silver pentafluorobenzoate (**22**) is blocked from decomposing to the acid by the absence of hydrogen. Only silver 3-phenylpropionate (**12**) of the compounds with both double bonds and hydrogen gives no evidence of decomposition.

A few of the salts show ions not containing silver which derive from the combination of two carboxylate radicals, but with the exception of silver pentafluorobenzoate (**22**) these ions have relative intensities below 2%. In silver pentafluorobenzoate (**22**) some sixty ions with the compositions C_nF_m ($n = 1$–18 and $m = 1$–14) are observed with the base peak at m/z 334 ($C_{12}F_{10}$) and more than half of the total ion current accounted for by dimer and trimer derived fragments ($n \geq 7$). Although the spectra show little or no dependence on temperature it is not possible to exclude the possibility that these ions are from thermal decomposition products.



Ion *a*



Ion *b*

Many of the salts show low intensity peaks for $[Na]^+$ at m/z 23 and at the mass for ion *a*, a monosodium analog of ion *b* which is often the most intense silver containing ion. The mass spectra of alkali metal carboxylates show analogous ions.[7]

The FD spectra of silver acetate (**1**) and silver *p*-chlorobenzoate (**24**) are unexceptional. The ions Ag_nX_{n-1} for X = acetate ($n = 1$–6) and X = *p*-chlorobenzoate ($n = 1$–4) are observed. Observation of such clusters with one more cation than anion have been reported for sodium acetate.[8]

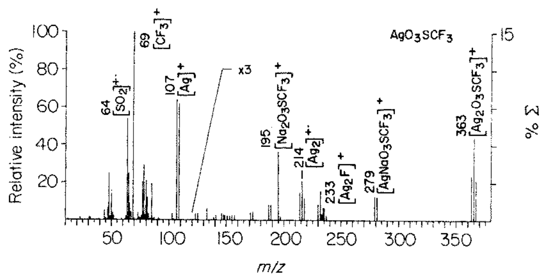


Figure 2. The 70 eV EI mass spectrum of silver trifluoromethanesulfonate (**27**). The corresponding sodium salt is present as a contaminant.

Silver compounds containing sulfur, phosphorus, or nitrogen

Of the six sulfur containing compounds examined (compounds **27**–**32** in Table 1) only the two fully fluorinated ones give EI spectra with silver containing ions. Silver trifluoromethanesulfonate (**27**) (Fig. 2) fragments very much like the silver carboxylates and apparently also vaporizes as a dimer as expected since an analogous cyclic dimer structure is possible.

The FD spectra of silver methanesulfonate (**28**) and silver *p*-toluenesulfonate (**29**) show the clusters $Ag_n(O_3SR)_{n-1}$ analogous to the carboxylates, but the size of the observable clusters is larger with $n = 1$–7 for **28** and $n = 1$–5 for **29**. Further information on the FD spectra of silver sulfonates will be reported elsewhere.

Silver trifluoromethanethiolate (**30**) (Fig. 3) gives a spectrum of quite different appearance with ions indicating the presence of aggregates at least as large as tetramer. The linear twofold coordination of silver when bonded to oxygen is known to change to a nonlinear arrangement[9] when the ligand is sulfur. The spectrum and the consideration of reasonable bond angles are both consistent with vaporization of **30** as tetramer with a cubane structure or as a cyclic 8-membered ring of alternating silver and sulfur atoms.

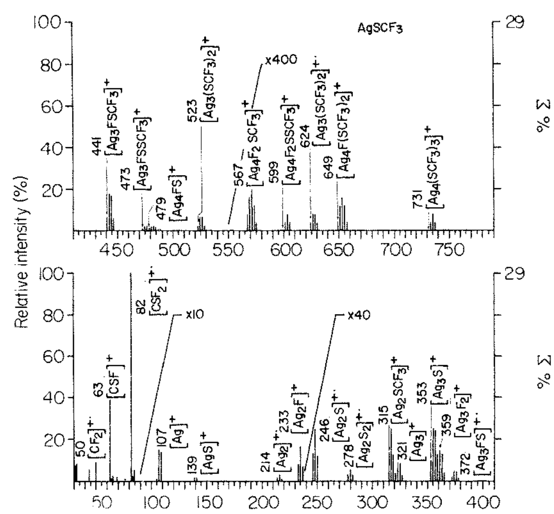


Figure 3. The 70 eV EI mass spectrum of silver trifluoromethanethiolate (**30**).

The absence of silver containing ions in the spectra of three sulfinates, silver methanesulfinate, benzenesulfinate, and *p*-toluenesulfinate, has been noted.[10]

The one phosphorus compound examined, silver diphenylphosphate (**33**) disproportionates and only the spectrum of tetraphenylpyrophosphate is observed.

Only the divalent silver compound, silver phthalocyanine (**34**) of the six nitrogen compounds (**11**, **23**, **26** and **34–36**) gives a spectrum with silver containing ions. A molecular ion at *m/z* 519 and silver ion at *m/z* 107 are present. Aside from the shift due to metal substitution, the spectrum is very similar to that of copper phthalocyanine.[11]

EXPERIMENTAL

Mass spectra

All mass spectra except as noted were recorded using a Finnigan 1015 mass spectrometer at 70 eV and a source temperature of 200 °C. Samples were introduced using a direct probe and gave intense spectra at source temperatures between 125 °C and 230 °C except for silver trifluoromethanesulfonate (**27**) and silver phthalocyanine (**34**) which required 388 °C and 393 °C respectively.

Metastable spectra and exact masses were determined on a Varian MAT CH-5 DF at 70 eV and a source temperature of 200 °C.

Field desorption spectra were determined on a Varian MAT CH-5 DF using emitters activated with benzonitrile.

Silver salts

Compounds **1**, **10**, **27**, **28**, **33**, **34** and **35** were commercial samples and were used as received except **10** which was recrystallized once from ether. Compounds **16** and **36** were available from sources in these laboratories and a sample of **30** was kindly provided by Dr J. Liu of these laboratories.

Compound **11** was prepared by adding *n*-heptafluorobutyric acid to a slight excess of silver oxide in ether with stirring. The excess silver oxide was removed by filtration and the product crystallized by partial evaporation of the ether.

Compound **29** was prepared from sodium *p*-toluenesulfonate and silver nitrate according to a published procedure.[12]

All other compounds were prepared from the corresponding sodium salt and silver nitrate using a modification of a published method.[13]

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