Object: Outgassing measurements of VAC-SEAL vacuum epoxy

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Vacuum compatible glues are to be used in different parts VIRGO, e. g. to fix the antispring magnets. We have investigated the behavior of the high vacuum sealant VAC-SEAL at various temperatures including the baking temperature of 150 ^{0}C. The test apparatus and the measurement method are described in detail in VACPISA025. The outgassing is measured using the dynamic method: measuring both pressures in the sample chamber p_1 and in the pumping chamber p_2 the total outgassing flow can be determined from the relation:

$$Q = (p_1 - p_2) \times C \quad (1)$$

where C is the conductance (20 l/s N_2 equivalent at 20 ^{0}C) between the chambers. The measurement is performed twice, with and without the sample in the chamber: the difference between the two flow values represents the net sample outgassing; the difference in the RGA spectra gives the mass distribution of the sample outgassing.

1 - System performances

The vacuum chamber has been pumped and baked at 200 ^{0}C for 3 days. The final performances were:

t(h)	p_1(mbar)	p_2(mbar)	Q(mbar l/s)
after baking	1.6×10^{-9}	4.0×10^{-10}	2.4×10^{-8}

The outgassing spectrum is shown in Fig. 1.

2 - Outgassing properties of VAC-SEAL

The VAC-SEAL is a two component epoxy manufactured by the Physical Electronics, US. The sample has been spread on a thin metal slab and cured at room temperature for 5 days. The exposed surface was 37.2 cm^2. We monitored the outgassing flow evolution (the time as measured from the beginning of the test will be used in the whole paper):

t(h)	T(^{0}C)	p_1(mbar)	p_2(mbar)	Q(mbar l/s)
0.25	35	2.4×10^{-5}	1.3×10^{-5}	2.2×10^{-4}
21	35	1.6×10^{-5}	9.2×10^{-6}	1.4×10^{-4}
42	35	8.8×10^{-6}	5.1×10^{-6}	7.4×10^{-5}
43.5	35	8.7×10^{-6}	5.1×10^{-6}	7.2×10^{-5}
48	35	1.4×10^{-5}	7.9×10^{-6}	1.2×10^{-4}

66	35	$6.0x10^{-6}$	$3.5x10^{-6}$	$5.0x10^{-5}$
71	35	$7.1x10^{-6}$	$3.8x10^{-6}$	$6.6x10^{-6}$
71.5	35	$8.5x10^{-6}$	$4.4x10^{-6}$	$8.2x10^{-5}$

The spectrum taken after 71.5 hours is shown in Fig. 2; we checked that the peak at 32 was not due a leak, it can be probably explained with the air trapped inside the epoxy. We set the temperature to 50 ^{0}C for 68 hours and monitored the evolution of outgassing flow:

t(h)	T(^{0}C)	p_1(mbar)	p_2(mbar)	Q(mbar l/s)
72	50	$6.4x10^{-5}$	$2.8x10^{-5}$	$7.2x10^{-4}$
72.5	50	$1.4x10^{-4}$	$6.1x10^{-5}$	$1.6x10^{-3}$
73	50	$4.2x10^{-5}$	$1.9x10^{-5}$	$4.6x10^{-4}$
89	50	$1.8x10^{-5}$	$1.0x10^{-5}$	$1.6x10^{-4}$
92	50	$1.7x10^{-5}$	$9.2x10^{-6}$	$1.6x10^{-4}$
95	50	$1.5x10^{-5}$	$7.9x10^{-6}$	$1.4x10^{-4}$
96	50	$1.1x10^{-5}$	$6.6x10^{-6}$	$8.8x10^{-5}$
98	50	$3.1x10^{-6}$	$2.3x10^{-7}$	$5.7x10^{-5}$
114	50	$1.6x10^{-6}$	$1.1x10^{-7}$	$3.0x10^{-5}$
115	50	$2.8x10^{-6}$	$1.6x10^{-7}$	$5.3x10^{-5}$
122	50	$1.4x10^{-6}$	$9.5x10^{-8}$	$2.6x10^{-5}$
138	50	$1.4x10^{-6}$	$8.5x10^{-8}$	$2.6x10^{-5}$
139	50	$8.1x10^{-7}$	$5.9x10^{-8}$	$1.5x10^{-5}$
140	50	$1.1x10^{-6}$	$7.8x10^{-8}$	$2.0x10^{-5}$

A spectrum taken after 68 hours at 50 ^{0}C is shown in Fig. 3. We set the temperature to 100 ^{0}C for 26.5 h and monitored the evolution of outgassing flow:

t(h)	T(^{0}C)	p_1(mbar)	p_2(mbar)	Q(mbar l/s)
141	100	$4.0x10^{-5}$	$4.6x10^{-6}$	$7.1x10^{-4}$
141.25	100	$2.5x10^{-5}$	$2.3x10^{-6}$	$7.7x10^{-4}$
143.25	100	$1.9x10^{-5}$	$6.6x10^{-7}$	$3.7x10^{-4}$
144.5	100	$2.0x10^{-5}$	$7.2x10^{-7}$	$3.9x10^{-4}$
146	100	$1.3x10^{-5}$	$4.3x10^{-7}$	$2.5x10^{-4}$
146.5	100	$9.9x10^{-6}$	$3.5x10^{-7}$	$1.9x10^{-4}$
163	100	$7.6x10^{-7}$	$5.6x10^{-8}$	$1.4x10^{-5}$
166.5	100	$9.0x10^{-7}$	$5.1x10^{-8}$	$1.7x10^{-5}$

A spectrum after a few hours at 100 ^{0}C is shown in Fig. 4 where there is contamination from hydrocarbon fragments (masses 41, 43, 45, 55, 57) and an unrecognized peak at mass 73. We set the temperature to 150 ^{0}C for 49 hours and monitored the evolution of outgassing flow:

t(h)	T(^{0}C)	p_1(mbar)	p_2(mbar)	Q(mbar l/s)
167	150	$9.2x10^{-5}$	$2.9x10^{-6}$	$1.8x10^{-3}$
167.5	150	$3.3x10^{-5}$	$1.1x10^{-6}$	$6.4x10^{-4}$
168	150	$2.2x10^{-5}$	$7.7x10^{-7}$	$4.2x10^{-4}$
169.5	150	$1.8x10^{-5}$	$6.2x10^{-7}$	$3.5x10^{-4}$
194.5	150	$3.1x10^{-6}$	$1.6x10^{-7}$	$5.9x10^{-5}$
195	150	$5.1x10^{-7}$	$4.8x10^{-8}$	$1.0x10^{-5}$

195.5	150	$1.0x10^{-6}$	$5.7x10^{-8}$	$1.9x10^{-5}$
196	150	$3.5x10^{-6}$	$1.7x10^{-7}$	$6.7x10^{-5}$
209.5	150	$1.8x10^{-6}$	$1.0x10^{-7}$	$3.4x10^{-5}$
215.5	150	$1.8x10^{-6}$	$1.0x10^{-7}$	$3.4x10^{-5}$

A spectrum taken at beginning of heating is shown in Fig. 5, which shows a huge organic contamination. We switched off the heating and monitored the evolution of outgassing flow:

t(h)	$T(^0C)$	p_1(mbar)	p_2(mbar)	Q(mbar l/s)
216	97	$3.8x10^{-7}$	$3.7x10^{-8}$	$6.9x10^{-6}$
216.5	86	$1.6x10^{-7}$	$2.2x10^{-8}$	$2.8x10^{-6}$
217.5	55	$4.7x10^{-8}$	$6.6x10^{-9}$	$6.7x10^{-7}$
233	28	$3.2x10^{-9}$	$1.5x10^{-9}$	$3.4x10^{-8}$
234.5	28	$9.2x10^{-9}$	$2.0x10^{-9}$	$1.4x10^{-7}$
237	28	$3.3x10^{-9}$	$1.3x10^{-9}$	$4.0x10^{-8}$
240	25	$5.9x10^{-9}$	$1.7x10^{-9}$	$8.4x10^{-8}$
258	25	$2.4x10^{-9}$	$1.2x10^{-9}$	$2.4x10^{-8}$
263	25	$2.5x10^{-9}$	$1.1x10^{-9}$	$2.8x10^{-8}$
281	25	$1.8x10^{-9}$	$7.6x10^{-10}$	$2.1x10^{-8}$

The spectrum taken after 281 hours is shown in Fig. 6.

We have summarized the evolution of the outgassing rate in Fig. 7.

3 - Conclusions

The VAC-SEAL epoxy resin achieved a final outgassing rate of $5.6x10^{-10}$ mbar l/(s cm^2) after baking. The outgassing composition at room temperature is quite good; we remind that with TorrSeal hydrocarbon contamination was always present. At temperatures above 100 ^0C there is emission of several hydrocarbon fragments which forbids vacuum baking in situ. We conclude that VAC-SEAL can be recommended for use in the top part of the towers. Despite the low outgassing rate, the use of any glue in the lower part of the tower should however be avoided.

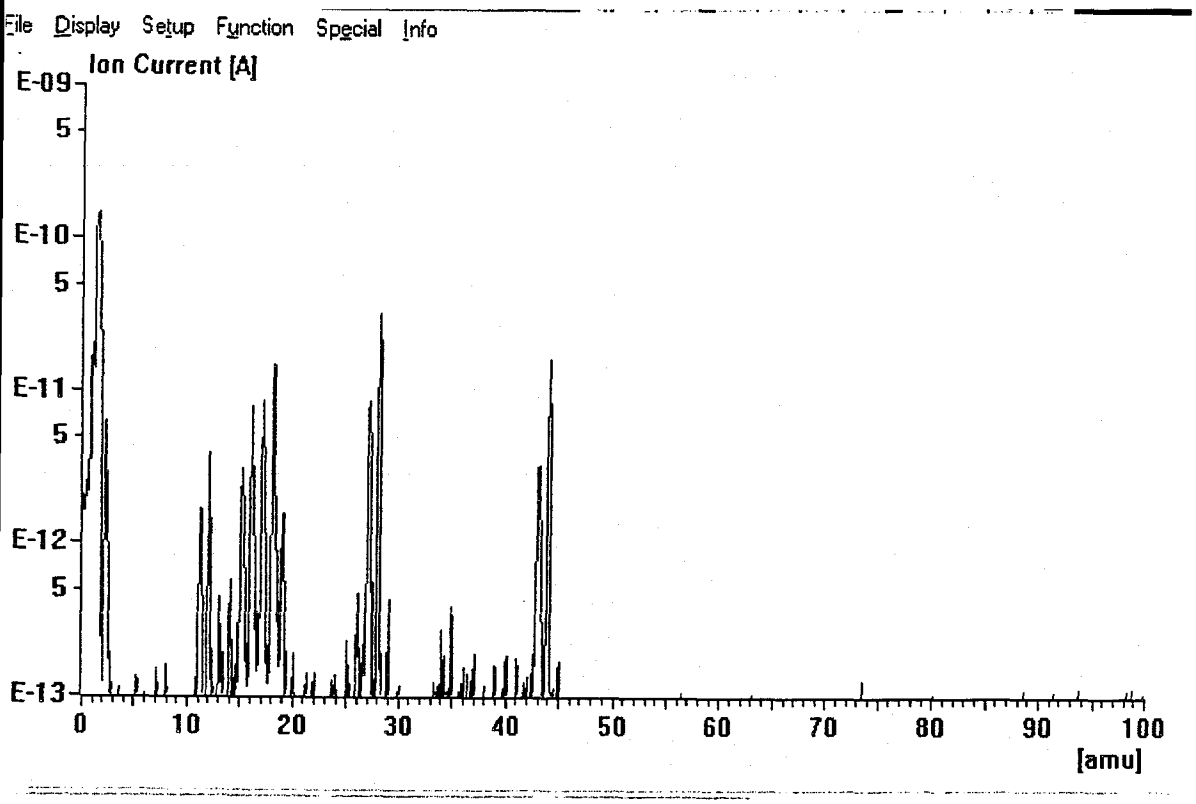

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Fig. 1

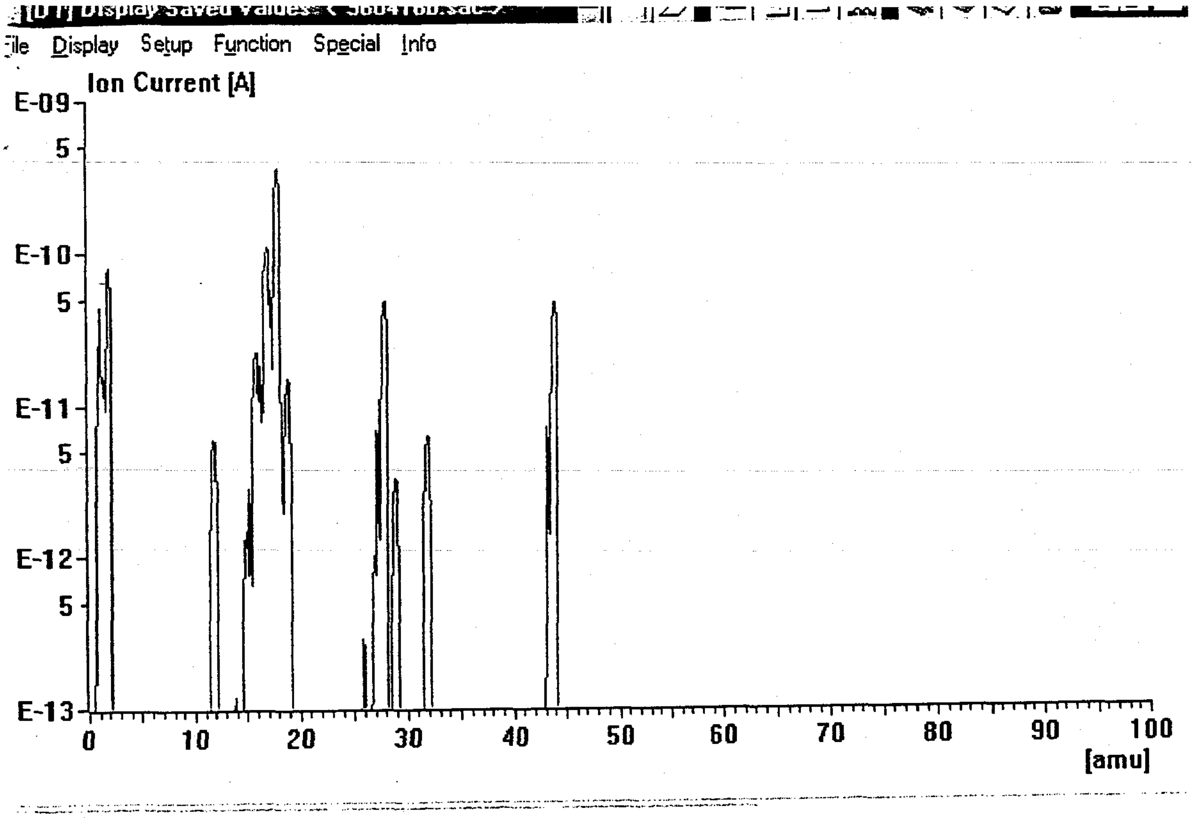
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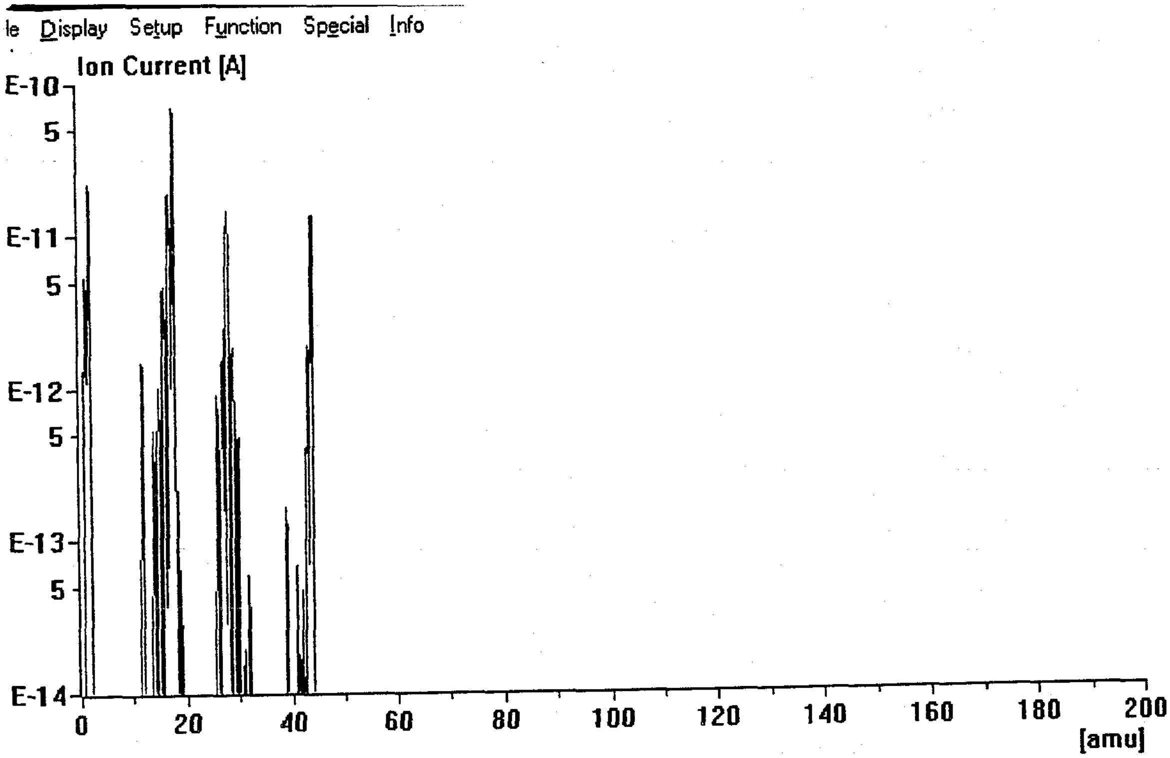
Fig. 2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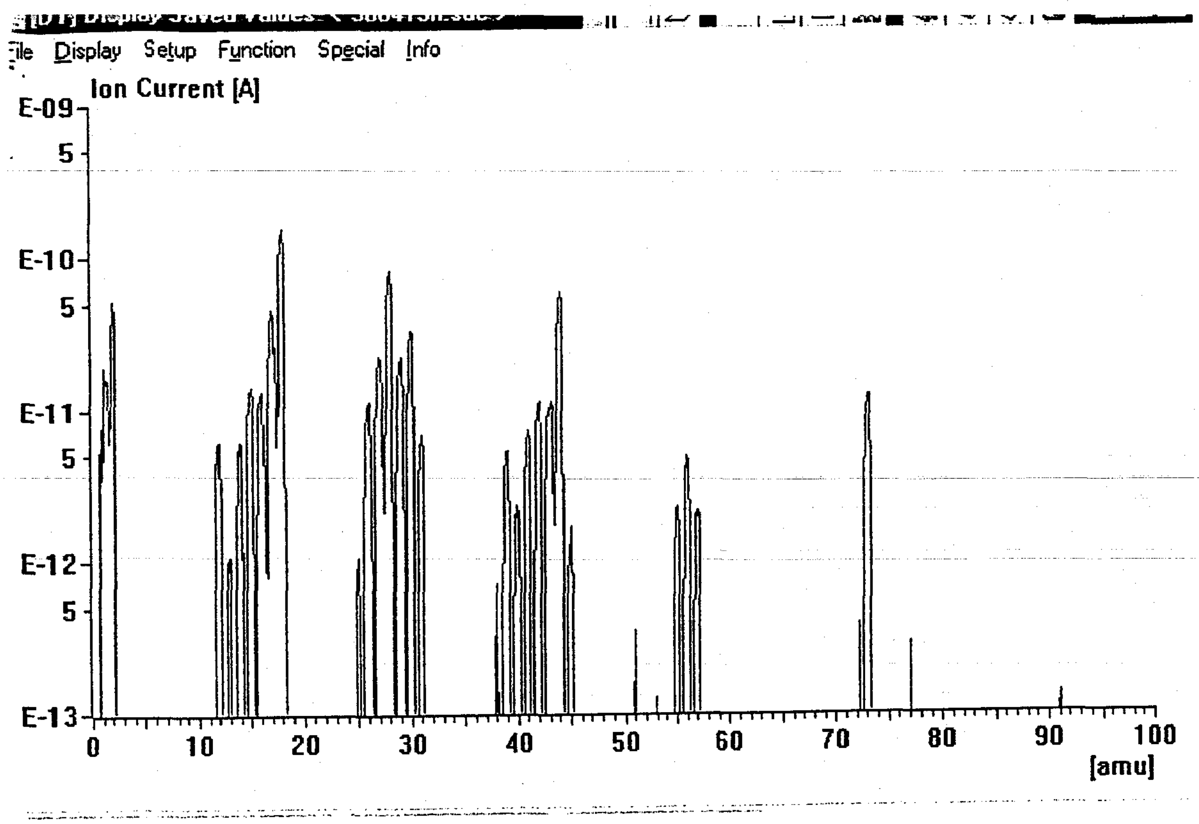
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Fig. 4

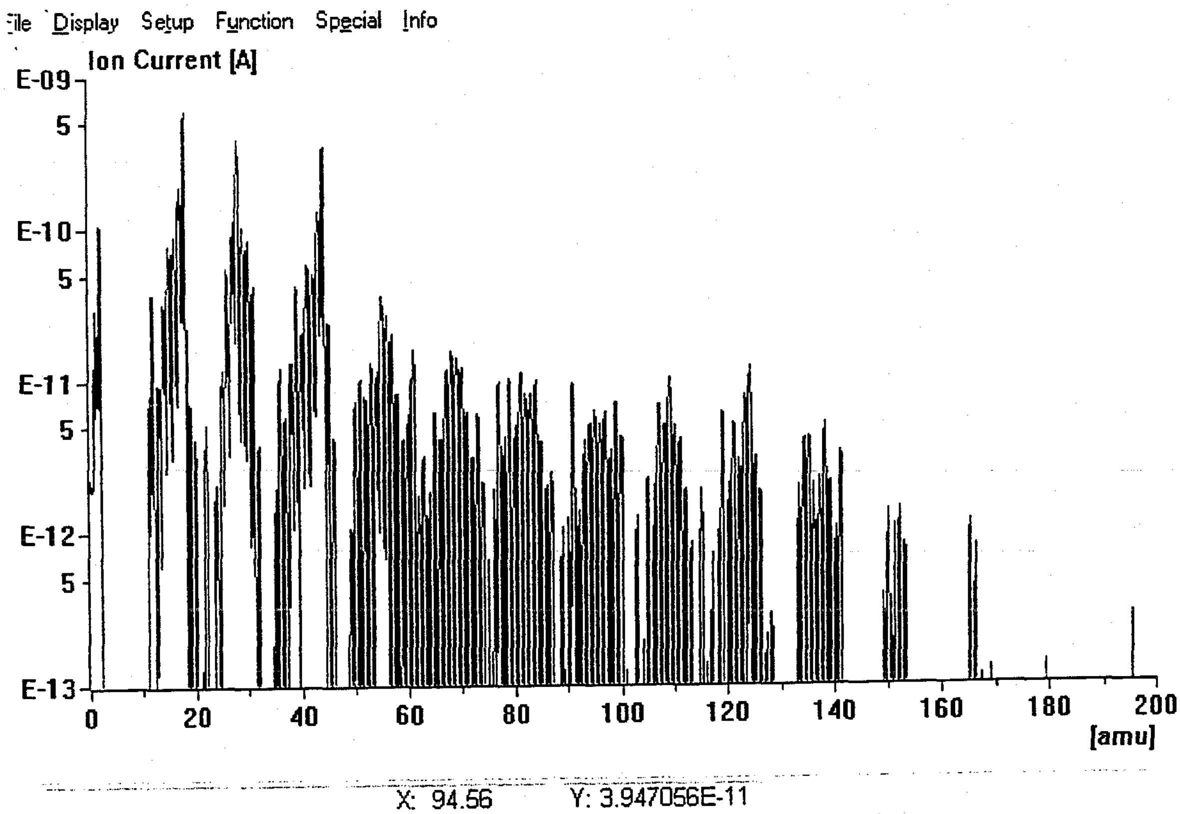
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Fig. 5

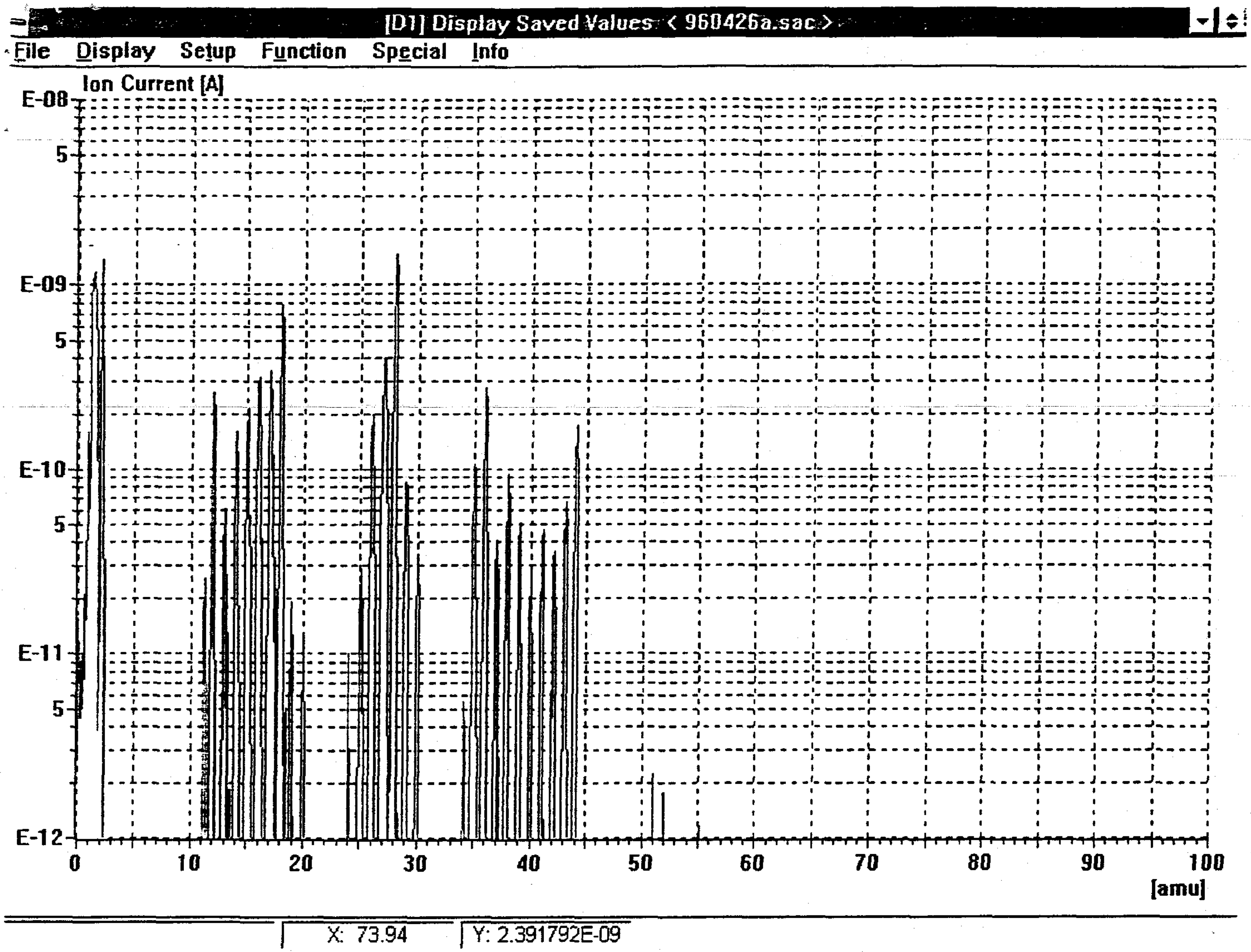

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Fig. 6

Sheet2 Chart 3