Object: Outgassing measurements of Krytox LVP grease

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In this note we briefly report the results obtained on Krytox LVP grease high vacuum grease by Dupont in view of its possible use as lubricant in the vacuum environment of VIRGO. The test apparatus is described in detail in the note VACPISA 025. 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 deduced from the relation:

$$Q = (P_1 - P_2) \times C \qquad (1)$$

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

1 - System performances

After baking the base pressure of the chamber is of the order of 10^{-9} mbar and the outgassing rate of the order of 10^{-11} mbar l s^{-1} cm^{-2}. The main components of outgassing are H_2, H_20, N_2/CO, CO_2.

2 - Measurement of the outgassing flow of Krytox

The Krytox LVP is a perfluorinated grease manufactured by Dupont whose main properties are summarized in Fig. 1, a). The behaviour of vapour pressure versus the temperature is shown in Fig. 1, b). The Krytox has been spread over a clean thin metal slab. The surface exposed to vacuum was 100 cm^2.

We performed a first test monitoring the outgassing evolution:

t(h)	T(^{0}C)	p_1(mbar)	p_2(mbar)	Q(mbar l/s)
1	12	3.4×10^{-5}	2.0×10^{-5}	2.8×10^{-4}
65	13	3.0×10^{-6}	1.5×10^{-6}	3.0×10^{-5}
70.5	13	6.1×10^{-7}	5.0×10^{-8}	1.1×10^{-5}
72	13	5.2×10^{-7}	4.1×10^{-8}	9.6×10^{-6}
73	13	4.7×10^{-7}	3.7×10^{-8}	8.8×10^{-6}
90	13	2.9×10^{-7}	2.0×10^{-8}	5.4×10^{-6}
97	12	3.1×10^{-7}	1.8×10^{-8}	5.9×10^{-6}

The outgassing after 97 h is reported in Fig. 2. There is an evident peak at mass 69 which has been recognized as CF_3. We stopped to start a test with a new sample. After baking we got:

t(h)	T(^{0}C)	p_1(mbar)	p_2(mbar)	Q(mbar l/s)
after	13	2.8×10^{-9}	6.4×10^{-10}	4.3×10^{-8}

The main component of outgassing were H_2, H_20, N_2/CO, CO_2.
We have inserted a new sample of Krytox with the same surface as the precedent one and monitored the outgassing evolution:

t(h)	T(^{0}C)	p_1(mbar)	p_2(mbar)	Q(mbar l/s)
8	22	4.8×10^{-7}	1.6×10^{-7}	6.4×10^{-6}
23	22	9.6×10^{-8}	9.8×10^{-9}	1.7×10^{-6}
30	22	9.1×10^{-8}	9.5×10^{-9}	1.6×10^{-6}

The spectrum after 30 h is reported in Fig. 3. We have heated the chamber at 35^{0}C inside for 2 days:

t(h)	T(^{0}C)	p_1(mbar)	p_2(mbar)	Q(mbar l/s)
during	35	8.1×10^{-8}	7.3×10^{-9}	1.5×10^{-6}

The spectrum measured after some hours is shown in Fig. 4, where we have identified C_2F_4 and C_2F_5 in addition to CF_3 and other high mass compounds.

We have heated the chamber at 49 ^{0}C for 3 days:

t(h)	T(^{0}C)	p_1(mbar)	p_2(mbar)	Q(mbar l/s)
during	49	6.0×10^{-8}	6.1×10^{-9}	1.1×10^{-6}

In the spectrum taken after 73h and reported in Fig. 5 we have identified some peaks corresponding to fluorine compounds: CF_3, C_2F_3O, C_2F_3, C_2F_5.

The time evolution of the outgassing rate is presented in Fig. 6.

3 - Discussion

We summarize here the outgassing rates measured in different conditions for Krytox LVP grease:

- temperature 22^0C, 30h pumping : $1.6x10^{-8}$ mbar l s^{-1} cm^{-2}

- temperature 35^0C, 46h pumping : $1.5x10^{-8}$ mbar l s^{-1} cm^{-2}

- temperature 49^0C, 73h pumping : $1.1x10^{-8}$ mbar l s^{-1} cm^{-2}

In all cases we observed at least one fluorine compound appearing in the outgassing spectrum. For this reason, we do not recommend Krytox LVP and more generally perfluorinated greases or oils such as Fomblin for use in Virgo. Solutions involving solid lubricants such as MoS_2, BN, SiN, TiN are going to be investigated in the near future.

Fig. 1

a)

Technical Information

Table 3
Krytox LVP High-Vacuum Grease*

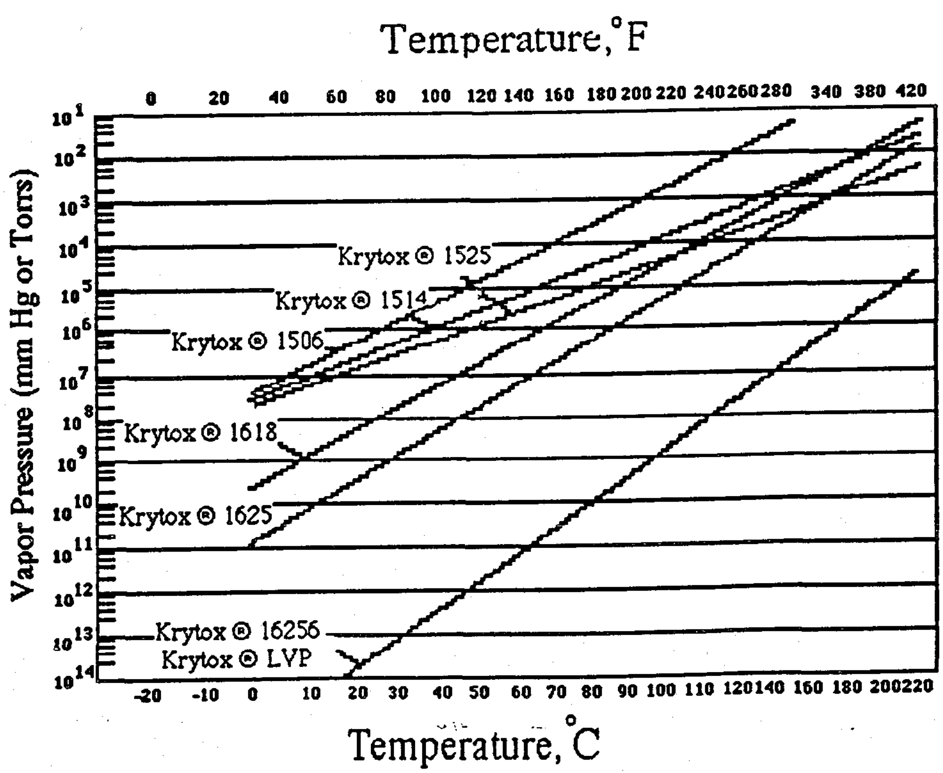
Penetration (worked, 25°C), mm/10	280
NLGI Consistency Grade**	2
Vapor pressure,***	
torr at 20°C	$\leq 1.0 \times 10^{-13}$
torr at 200°C	$\leq 1.0 \times 10^{-5}$
kPa at 20°C	$\leq 1.3 \times 10^{-14}$
kPa at 200°C	$\leq 1.3 \times 10^{-6}$
Oil Separation, (30 hrs, 204°C), wt%	13.8
Evaporation Loss, (22 hrs, 204°C), wt%	0.3
Density, (25°C), g/cm3	1.94

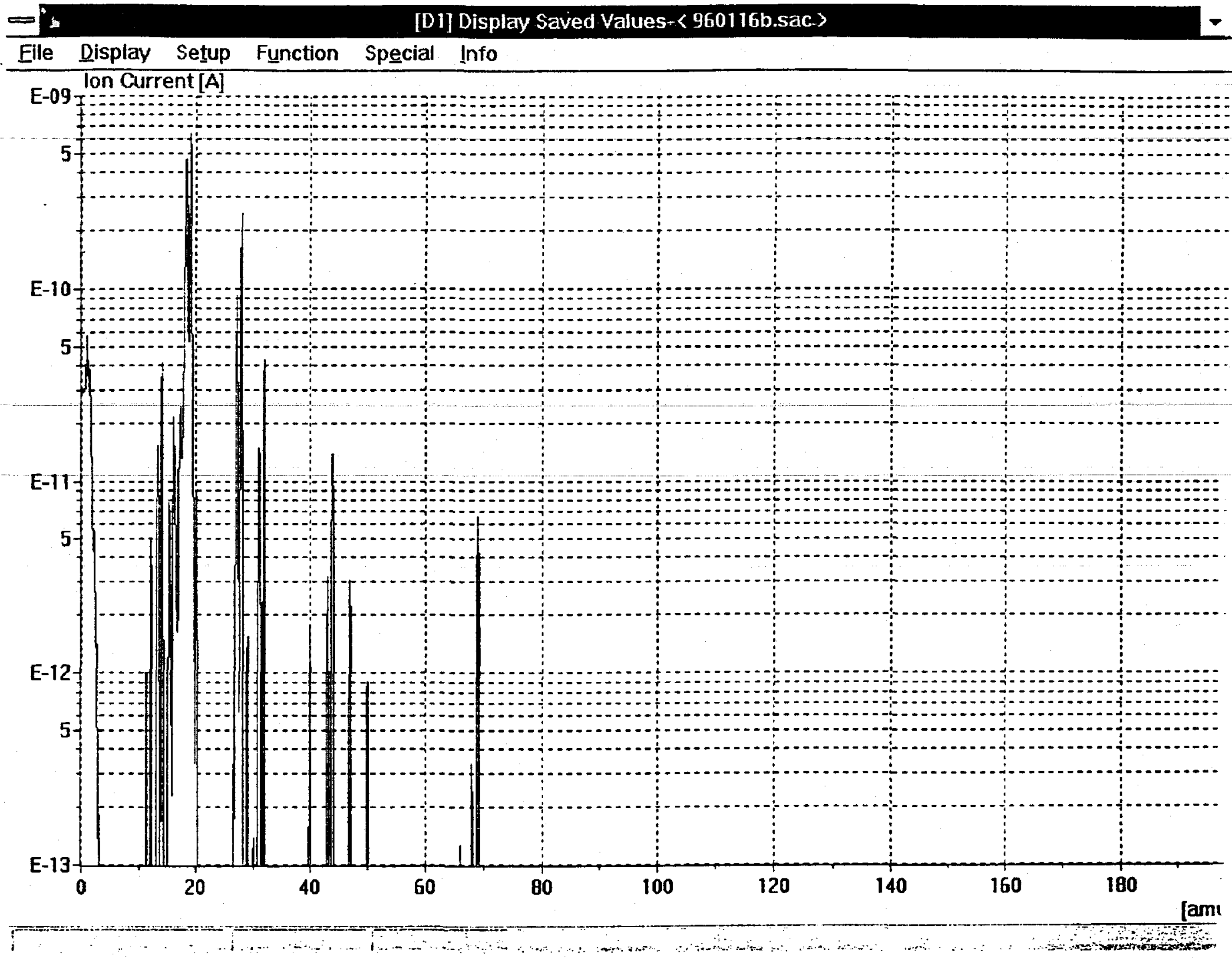
*This table gives typical properties based on historical production performance. DuPont does not make any express or implied warranty that these products will continue to have these typical properties.

b)

Technical Information

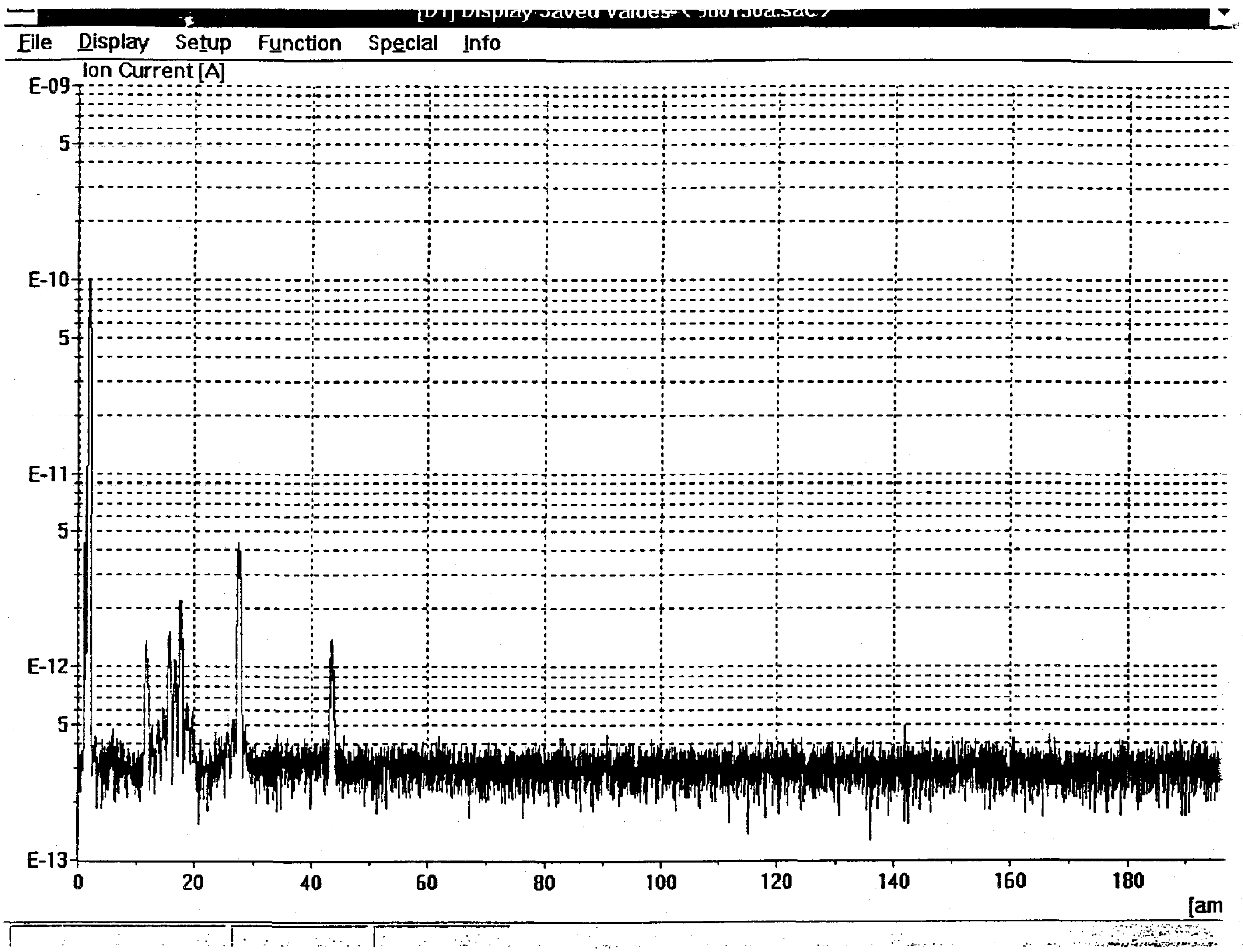
Typical Vapor Pressure-Temperature Characteristics





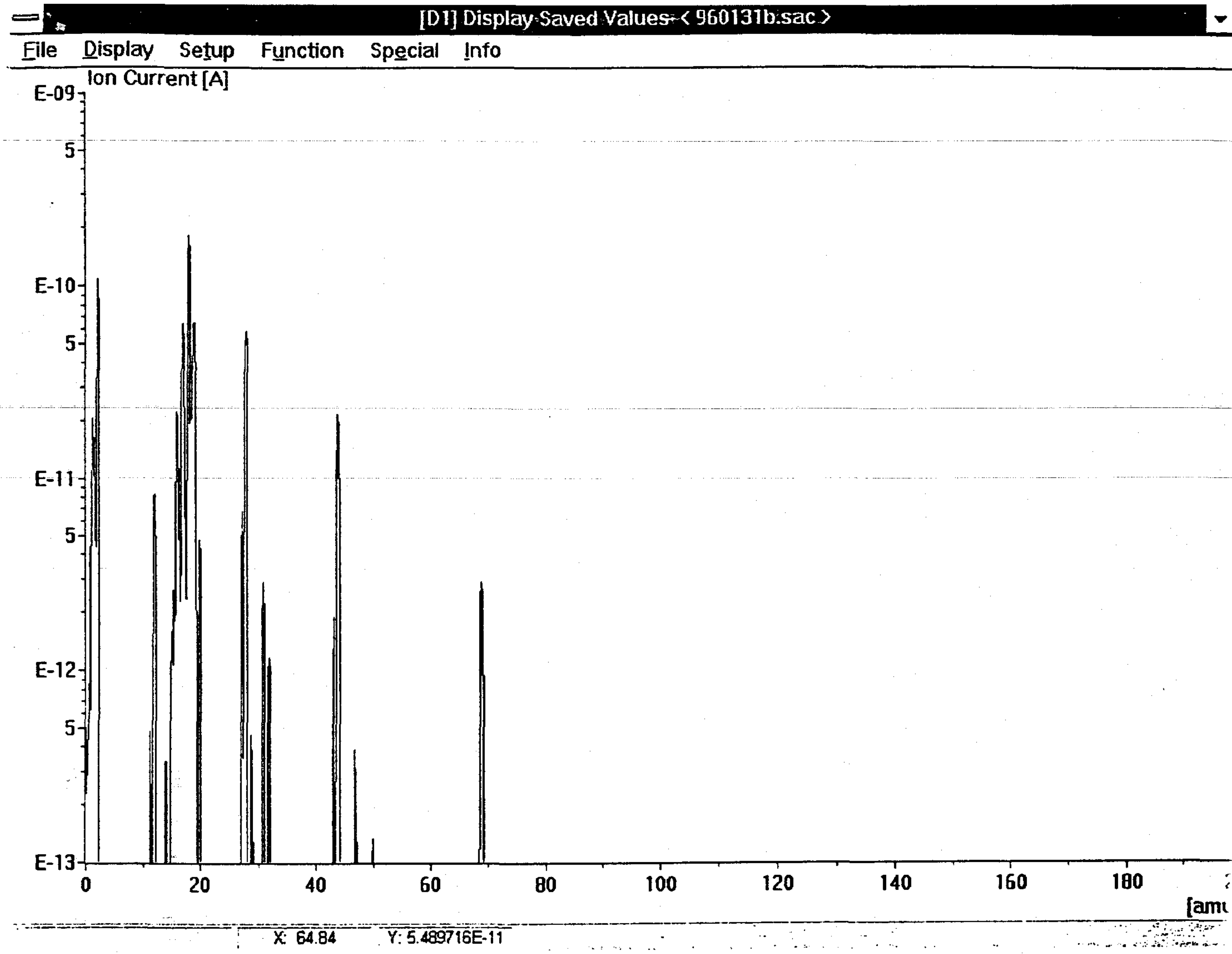
$p = 3.1 \times 10^{-7}$ mbar

Fig. 2



$p = 2.8 \times 10^{-9}$ mbar

Fig. 3



$p = 3.1 \times 10^{-8}$

Fig. 4

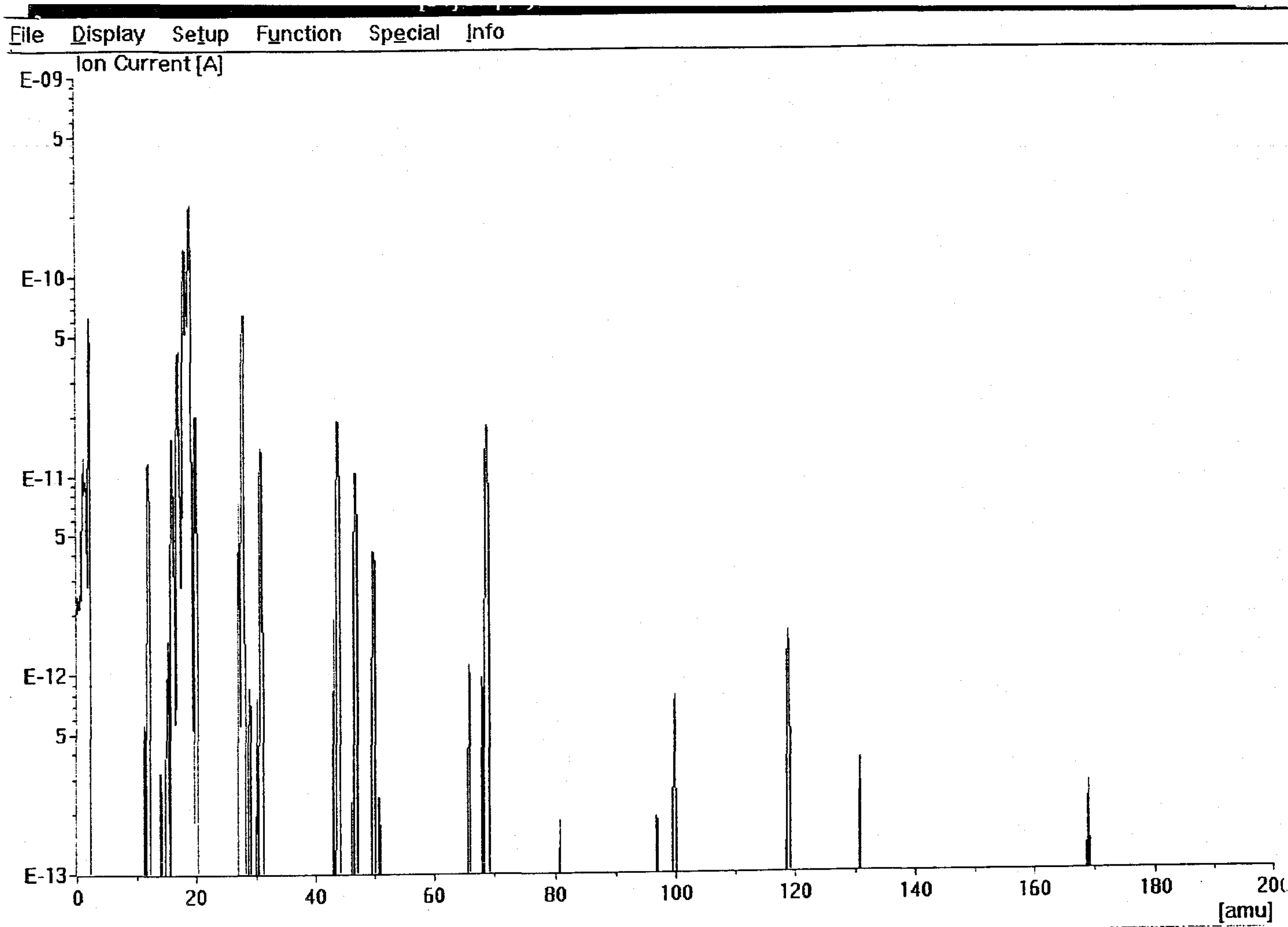

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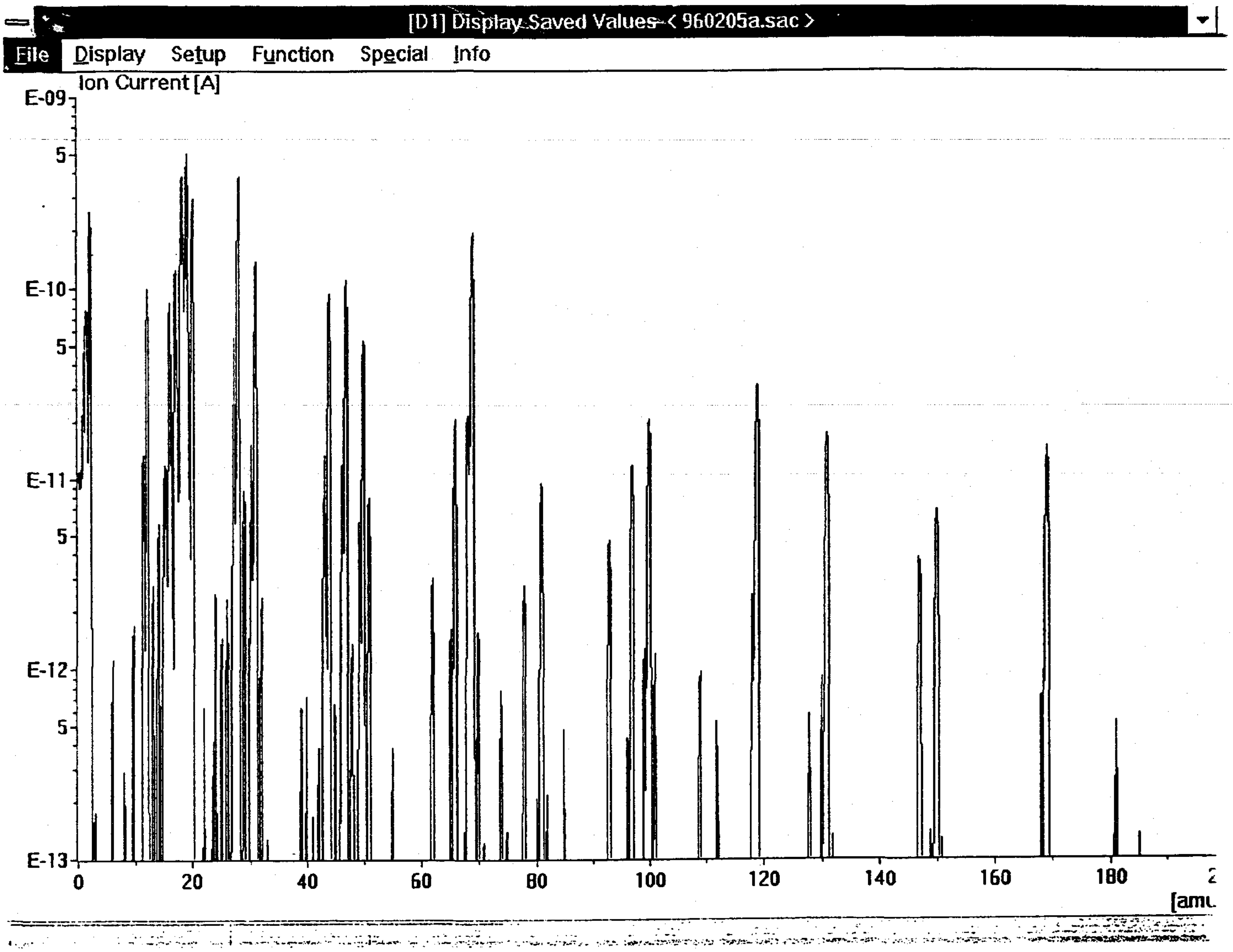
Fig. 5



$p = 6.0 \times 10^{-8}$ mbar

Fig. 6

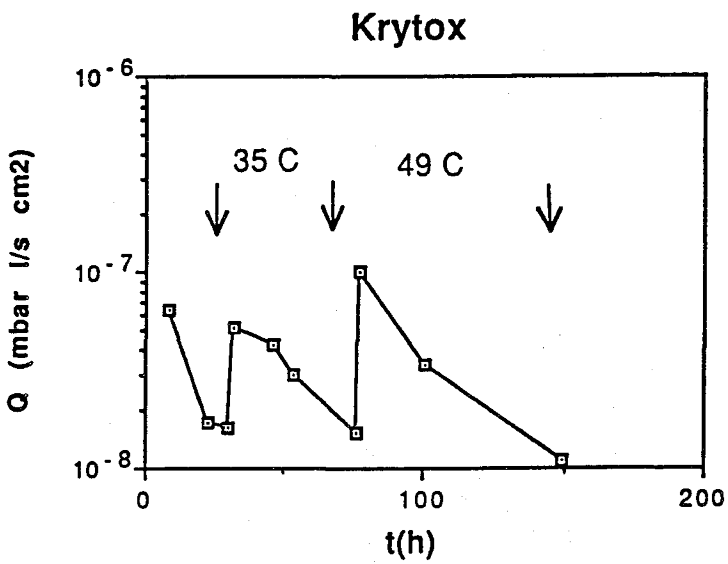


Fig.7