

Chromatographic study of element 104 in the system HCl/Tributylphosphate (TBP)

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Early experiments with element 104 showed that its chemical properties are analogous to those of the group-4 elements Zr and Hf [1,2]. More recently, the effects of HCl, hydrogen and chloride ion concentration on the extraction of element 104 into TBP/benzene were studied in liquid-liquid extraction experiments [3]. It was found that in 8 to 12 M HCl the extraction of element 104 is between that of Zr and Hf. The reported low extraction yields of Hf [3] could not be reproduced by us and are also being challenged by Kacher et al. [4] who reported that the extraction decreased in the order $Zr > Hf > 104 > Ti$. Therefore, it was of interest to reexamine the extraction behaviour of element 104 relative to that of its homologs in another, independent experiment.

The new experiments with the Automated Rapid Chemistry Apparatus ARCA II [5] were performed with pure TBP attached onto a inert support (Voltalef 30-80 μm , weight ratio TBP:Voltalef = 1:5) filled into the chromatographic columns of 1.6x8 mm size. Using these columns and carrier-free ⁹⁸Zr (from fission) and ¹⁶⁹Hf (from the reaction of ¹⁸O with ^{nat}Gd) a separation of Zr from Hf could be achieved as follows, see Fig. 1. Zr and Hf were loaded onto the column in 12 M HCl. Subsequently, a Hf fraction was eluted in 200 μl of 8 M HCl (containing 75 % of the Hf and no Zr), followed by the stripping of the Zr with 167 μl of 2 M HCl (containing the remainder of the Hf and >93 % of the Zr). ²⁶¹104 produced at the the Philips Cyclotron at PSI in the ²⁴⁸Cm(¹⁸O,5n) reaction, and transported by a He/KCl-jet to ARCA, was subject to the same chromatographic separation. After evaporation to dryness on Ta disks, the samples were measured by α -spectroscopy. In the α -energy range 8.0 to 8.65 MeV, 20 α -decays attributed to ²⁶¹104 and its daughter ²⁵⁷No were recorded, among those 5 mother-daughter correlations. Monte Carlo simulations of the random correlation rate showed that these are true correlations. From the distribution of the α -events between the Hf fraction (16) and the Zr fraction (4) we can calculate the distribution coefficient for element 104 for 8 M HCl/TBP, $K_d = 124_{-83}^{+133}$, which is of the same order of magnitude as the value for Hf which is 65, see Fig. 2. Thus, we find that the extraction from 8 M HCl into pure TBP decreases in the order $Zr > 104 \geq Hf$. Our data do not corroborate a lower extraction of element 104 than that of Hf [4] which was tentatively attributed to a stronger hydrolysis of element 104 than of Zr and Hf [4].

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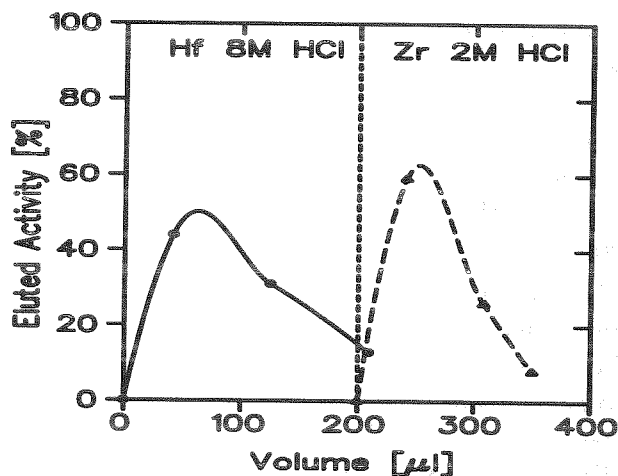


Fig.1: Separation of Hf and Zr on a 1.6x8 mm TBP/Voltalef column in ARCA II

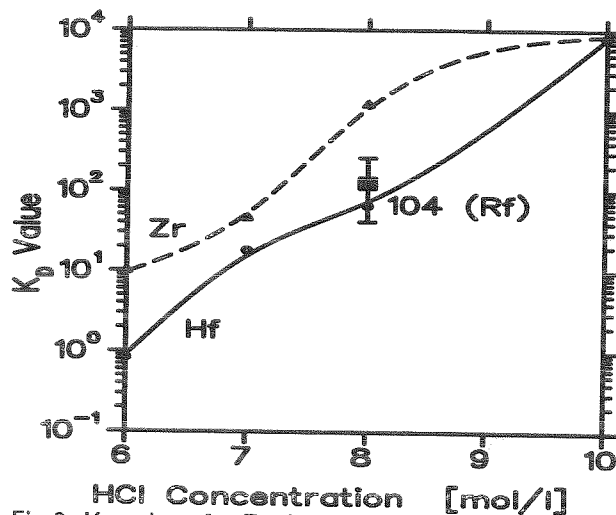


Fig.2: K_d values for Zr, Hf, and the element 104 in the system HCl/TBP

First Thermochemical Property of Seaborgium Determined

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The first successful experiments on the chemistry of element 106, seaborgium (Sg), [1] showed that Sg forms hexavalent ions in aqueous solution which form compounds similar to the ones formed by its lighter homologs in group 6 of the Periodic Table, Mo and W [2], and that Sg forms volatile oxychlorides [3]. The isotopes ^{265}Sg and ^{266}Sg were separated and unambiguously identified with the On-Line Gas Chemistry Apparatus, OLGA III [3]. This contribution reports first results from our second experiment to determine the volatility of Sg-oxychlorides in respect to their lighter homologue group-6 oxychlorides, and to obtain more information on the nuclear decay properties of ^{265}Sg and ^{266}Sg .

Relativistic calculations [4] showed that SgO_2Cl_2 should be the most stable oxychloride compound of Sg and from calculated overlap population data and dipole moments it was predicted that SgO_2Cl_2 should be the least volatile MO_2Cl_2 compound ($M = \text{Mo}, \text{W}, \text{Sg}$). Extrapolations of thermochemical properties yielded an adsorption enthalpy of $\Delta H_a^\circ(\text{SgO}_2\text{Cl}_2) = -106 \text{ kJ/mol}$ [5].

First, the volatility of WO_2Cl_2 was measured under identical conditions to the Sg experiment, and its yield-versus-temperature curve, see e.g. [1, 3], was found in good agreement with the previous measurement. Sg isotopes, produced at 123 MeV ^{22}Ne on ^{248}Cm , were transported with a He/C-jet to OLGA III where 144 ml/min Cl_2 , 20 ml/min SOCl_2 and 2 ml/min O_2 were added as reactive gases. Volatile products which passed through the chromatography were attached to aerosols and were transported to the Rotating Wheel Multidetector Analyser for spectroscopy of α -particles and fragments from Spontaneous Fission (SF). Every 10 s, the wheel was stepped to move the collected activity between 7 pairs of PIPS detectors. Simultaneously, a HPGe-detector was used to monitor the chemical yield by using short-lived W-isotopes produced from small amounts of ^{152}Gd in the ^{248}Cm target.

At 350°C isothermal temperature and with a beam dose of 2.31×10^{17} ^{22}Ne ions two decay chains attributed to ^{265}Sg and one decay chain attributed to ^{266}Sg were observed. This confirmed our first ex-

periment at which Sg decay chains were observed at 300°C and 400°C isothermal temperature. At a lower temperature of 250°C a beam dose of 5.22×10^{17} ions was accumulated without observing any $^{265,266}\text{Sg}$ decay chain. However, ^{168}W was still detected with a yield of 75 % relative to the yield at 350°C.

Together with the decay chains observed in the previous experiment [1, 3], and one ^{266}Sg α -SF decay correlation from a collection on tape without chemistry, more accurate half-lives were established (68 % c.i.) for ^{265}Sg , $T_{1/2} = (5.4 + 4.4 / - 1.7) \text{ s}$, and ^{266}Sg , $T_{1/2} = (20.8 + 28.4 / - 7.6) \text{ s}$.

The observed zero events at 250°C is in full agreement with the predicted volatility of SgO_2Cl_2 . In order to compare the volatility of SgO_2Cl_2 with its lighter homologs, the limits of possible adsorption enthalpies have to be determined. They are given by a relative yield of $\leq 32\%$ (from the statistical error of 0 events observed) at 250°C, a lower half-life limit of 3.7 s, a relative yield of $\geq 62\%$ at 350°C, and an upper half-life limit of 49.2 s. The corresponding interval of adsorption enthalpy is:
 $-95 \text{ kJ/mol} \leq \Delta H_a^\circ(\text{SgO}_2\text{Cl}_2) \leq -130 \text{ kJ/mol}$.

Therefore, the relative volatility of group 6 oxychlorides is $\text{MoO}_2\text{Cl}_2 > \text{WO}_2\text{Cl}_2 \geq \text{SgO}_2\text{Cl}_2$. The error limits on the adsorption enthalpy depend on the statistics of the experimental data to observe a Sg isotope and on the error limits of the half-life. More accurate half-life measurements will significantly reduce the current interval of possible adsorption enthalpy values. Nevertheless, we have determined the first thermochemical property of a compound of the heaviest element currently accessible by chemical means.

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Electronic Structure of the Hexahydrated Pentavalent Nb, Ta and Ha

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Recent experiments¹ on the extraction of Nb, Ta and Pa (as analogs of element 105, Ha) at tracer concentrations from the pure HCl and HBr solutions into an organic phase (a quaternary amin) have shown a much weaker extraction of Ta in comparison with the other elements. Such a behavior of Ta has also been observed earlier under other experimental conditions.² The reason for the weak extraction of Ta was thought to be strong hydrolysis, preventing Ta from the complex formation. Thus, the problem of hydrolysis was realized to be a key point in the process of extraction of these elements. To understand and predict (for Ha) the hydrolysis of group 5 elements, the present study has been undertaken.

At the initial step of it the calculations of the hydrated complexes of Nb, Ta and Ha have been performed using the Dirac-Slater Discrete-Variational (DS DV) method. The geometric configuration of $\text{Ha}(\text{H}_2\text{O})_6^{5+}$ has been assumed by analogy with those of Nb and Ta, having a regular octahedron surrounding of oxygen atoms and belonging to the T_h symmetry group (Fig. 1).

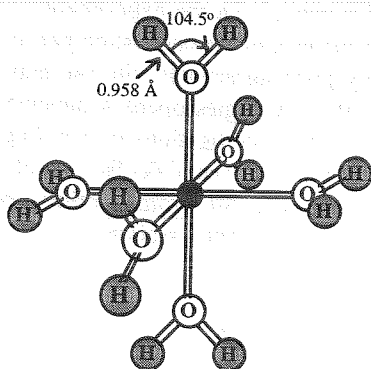


Fig. 1. The hexahydrated complex in T_h symmetry.

The energies of the highest occupied molecular orbital (HOMO), the lowest unoccupied MO (LUMO), the energy gap, ΔE , between them and other unoccupied levels of d-character, obtained as a result of calculations are given in Table 1.

Table 1. Energies of the HOMO, LUMO, ΔE (eV) for $\text{M}(\text{H}_2\text{O})_6^{5+}$, where $\text{M}=\text{Nb}$, Ta and Ha

MO	Nb	Ta	Ha
6g, 7g	-25.96	-24.75	-23.47
5g	-30.26	-29.35	-28.10
7g, 6g (LUMO)	-30.40	-29.82	-29.23
6u, 7u (HOMO)	-33.87	-33.88	-33.48
ΔE	3.47	4.06	4.25

Table 2. Effective charges (Q_A), energies of the Coulomb interaction, E^C , and total overlap populations (OP) for $\text{M}(\text{H}_2\text{O})_6^{5+}$, where $\text{M}=\text{Nb}$, Ta and Ha

Parameter	Nb	Ta	Ha
Q_M	2.15	2.23	2.20
Q_O	-0.66	-0.68	-0.68
Q_H	0.56	0.57	0.57
E^C , eV	14.37	17.46	15.68
OP(tot)	6.95	7.33	7.51

The electronic density distributions are presented in Table 2. The character of the metal-water bonding is proved to be highly ionic. An increase in the both ionic E^C and covalent (OP) contributions to bonding are indicative of the fact that the strength of the metal-water interaction increases from Nb to Ta, with the one for Ha being of the order of that for Ta. Hahnium has the strongest covalent bonding with the water molecules which is accounted for by the strongest $7s(\text{Ha})-2p(\text{O})$, $6d_{3/2}(\text{Ha})-2p(\text{O})$ and $6d_{5/2}(\text{Ha})-2p(\text{O})$ interactions.

An increasing energy gap between HOMO and LUMO is a reason to think about a regular octahedron structure of the complexes under consideration. The tetragonal distortion of the Ha complex which could have been supposed due to a pseudo Jahn-Teller effect will not take place due to a large value of ΔE ($\Delta E > 2a^2/K$, where K is the force constant and $a=dV/dR_0$, with V being the molecular potential and R_0 an internuclear distance in the point of equilibrium). Thus, all the water molecules will be equally bounded to the Ha ion.

A stronger bonding of the water molecules in the Ta and Ha complexes could be a reason to think about their weaker hydrolysis in comparison with Nb. (The weaker hydrolysis of Ta is also indicated in ref. 3). The ionic theory also suggests a weaker hydrolysis of Ha due to a larger ionic radius of M^{5+} , though it can not explain the hydrolysis of Ta. In practice, the real electronic density distribution including changes both in the ionic and covalent contributions into bonding, should be taken into account for the reaction



Calculations of the hydrolyzed species of group 5 elements are in progress. Final estimates of the free energy of reaction (1) and reactions of complex formation will hopefully allow to predict the extraction behavior of Ha.

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Calculations of Dissociation Energies for Very Heavy Element Compounds

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Due to a recent improvement¹ in the DS DVM code of Rosen and Ellis, it became possible to calculate directly dissociation energies of molecules containing heavy atoms and a large number of ligands with sufficient accuracy.

The single-particle equations for the wave functions ϕ_i and energy eigenvalues ε_i are

$$\left[t + V^n + V^c + V^{ex} \right] \phi_i = \varepsilon_i \phi_i \quad i=1, \dots, N \quad (1)$$

The direct Coulomb potential V^c has been approximated by a potential \tilde{V}^c resulting from a model density

$$\rho(r) \cong \tilde{\rho}(r) = \sum_k d_k f_k(r) \quad (2)$$

which is given as a linear combination of spherical, symmetric functions $f_k(r)$ centered at the atoms of the molecule. The expansion coefficients are determined from an additional least-squares fit in which the error

$$\Delta = \iint d^3r d^3r' \frac{[\rho(r) - \tilde{\rho}(r)][\rho(r') - \tilde{\rho}(r')]}{|r - r'|} \quad (3)$$

in the total energy is minimized. The total energy is then

$$E = \sum_i \tilde{\varepsilon}_i - \frac{1}{2} [\tilde{\rho} \tilde{V}^c d^3r - \frac{1}{4} [\rho V^{ex} d^3r + \sum_{p>q} \frac{Z_p Z_q}{R_p - R_q}]] + \Delta \quad (4)$$

where $\tilde{\varepsilon}_i$ is obtained via replacing V^c by \tilde{V}^c in Eq. (1). If $\tilde{\rho}$ is determined such that Eq. (3) is minimized, one can show that replacing V^c by \tilde{V}^c in Eq. (1) yields a variationally consistent total energy from Eq. (4).

Another improvement is the calculation of the Fock and overlap matrix elements, H_{ij} and S_{ij} , respectively, with the integration scheme of Boerrigter *et al.*². In addition, a special transformation has been introduced to treat the singularity of the relativistic wave function at the origin; it is now possible to achieve high accuracy with a reasonable number of points (e.g., 10^8 with about 1400 integration points).

As one of the first results for the polyatomic very heavy element compounds we present here the data for the group 6 highest chlorides, MCl_6 , where $M=Mo, W$ and element 106, seaborgium (Sg). They are summarized in Tables 1 and 2 along with results of other calculations.

Table 1. Binding energies, D_e (eV), for MCl_6 ($M=Mo, W$ and Sg) obtained as a result of different calculations

Method	MoCl ₆	WCl ₆	SgCl ₆	Ref.
DS DVM	(19.29)	(21.65)	20.11	3
DS DVM	20.97	22.18	20.05	this
DF	6.73	11.49	12.71	4
exp.	19.29	21.65	-	5

Table 2. Equilibrium bond distances, R_e (Å), for MCl_6 ($M=Mo, W$ and Sg) obtained as a result of different calculations

Method	MoCl ₆	WCl ₆	SgCl ₆	Ref.
DS DVM	(2.254)	(2.26)	2.39	3
DS DVM	2.33	2.36	2.45	this
DF	2.25	2.31	2.38	4
exp.	2.254	2.26	-	5

The equilibrium bond distances and binding energies have been obtained as results of a Morse fit of the corresponding potential-energy curves. One of them, as an example, is presented in Fig. 1, showing a pronounced stability of SgCl₆ at 2.45 Å.

The new D_e and R_e values are in very good agreement with experiment (2-5% difference). One can see that the estimates obtained earlier³ are also well in line with the new results. D_e obtained by the all-electron *ab initio* Dirac-Fock (DF) method⁴ recover only 40-60% of the binding energy, though give better R_e . The large difference with the experiment of the DF results is accounted for the lack of the correlation part of the DF code.

Thus, the very good agreement with the experiment for the binding (dissociation) energies opens a perspective of the use of the program in the investigations of bonding in even more complicated compounds. The calculations of the electronic structure and bonding of some other compounds of rutherfordium, hahnium and seaborgium are in progress.

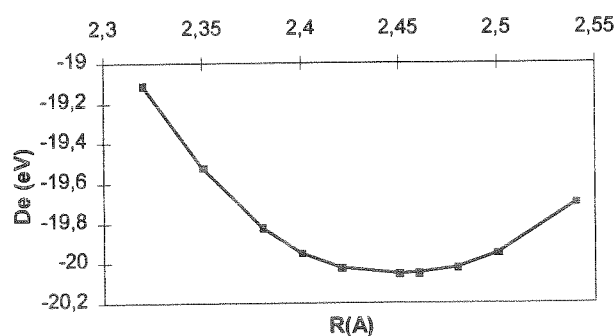


Fig. 1. Calculated potential energy curve for SgCl₆.

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Ion induced formation of colloids in LiF at 15 K

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In lithium fluoride and in other alkali halides, the formation and transformation of defects upon excitation of the electron subsystem has been studied for many years. The primary defects are F -centers and hole-interstitial H -centers. At a high dose, complex microdefects (aggregation to F_n and V_n -centers) and macrodefects (transformation of nF -centers to metallic Li colloids), covalent molecular anions (fluorine molecules), and vacancy clusters can be created [4, 5]. In earlier studies it has been demonstrated that only macroscopic colloids can be etched whereas evidence for the etchability of primary or microdefects has never been found. More recently, etchable tracks have been found when LiF was irradiated with heavy ions above an energy loss of about 1.2 keV/Å [3]. Using small-angle x-ray scattering (SAXS) and optical absorption spectroscopy, it has been shown that in a core of about 30 Å in diameter, macrodefects are formed, while primary defects such as color-centers (F and F_2) are created in an outer zone larger by up to one order of magnitude [4].

To shed more light on the mechanism of the creation of macrodefects along ion tracks, LiF crystals were irradiated at various low temperatures in the range between 15 K and room temperature. The selection of the irradiation temperatures was based on the fact that specific processes mandatory for defect creation are hindered at the following temperatures: At 15 K, the color centers of the primary Frenkel pairs are not mobile. Therefore, the probability for recombination processes is very high. At 90 K, the hole centers (H -centers) become mobile and form V_K -centers (self-trapped holes). At 150 K, V_K -centers are not stable, and primary H -centers are transformed to more complex hole centers (V_n -centers).

Thin platelets (0.5 mm) cleaved from a single-crystal block of high-purity LiF were irradiated under normal incidence with Pb ions of 5.9 MeV/u. To test the formation

of macrodefects, chemical etching was performed. Each single track was visualized by an etch pit of pyramidal shape owing to the cubic symmetry of the LiF lattice. Track etching was observed for all tested irradiation temperatures, giving clear evidence that the formation of etchable aggregates along ion tracks takes place even at a temperature as low as 15 K. Etching was also performed on samples which were cleaved parallel to the ion tracks (Fig. 1). The length of the etched structures is 40 μm and corresponds to the ion range where etchable aggregates are created.

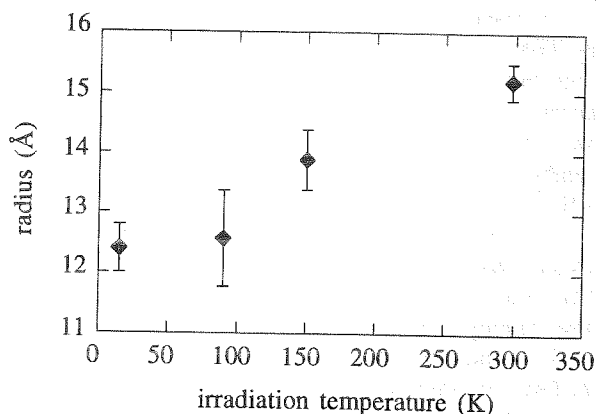


Fig. 2. Radius of ion tracks as a function of the irradiation temperature determined by small-angle x-ray scattering of 10^{10} Pb ions/cm² (5.9 MeV/u).

In order to determine the size of the macroscopic aggregates, SAXS experiments were performed on crystals irradiated with 10^{10} ions/cm². The scattering intensity was registered on a position sensitive area detector as a function of the scattering vector [5]. The radius of the colloids is only slightly influenced by the irradiation temperature (Fig. 2). The process of colloid formation at such low irradiation temperatures has never been observed when irradiating LiF with low ionizing radiation or gammas. It can only be understood if an increase of the local temperature possibly inducing a plasma-like state is assumed.

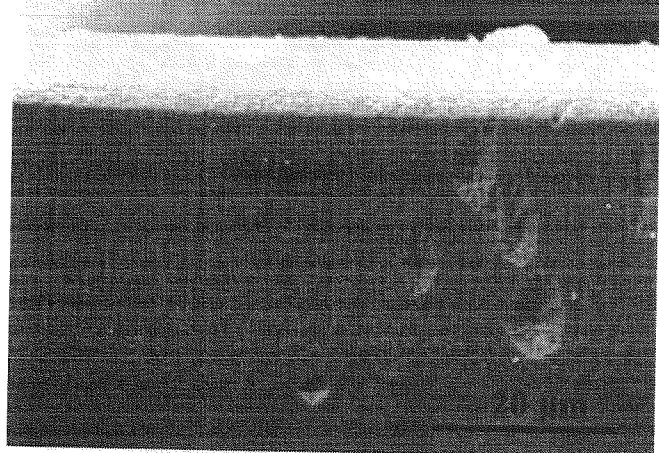


Fig. 1. Tracks of Pb ions (10^{10} cm⁻²) in a LiF crystal irradiated at 15 K and cleaved parallel to the ion beam after etching (20 min in a solution of concentrated HF (1 Vol) and CH₃COOH (1 Vol) saturated with FeF₃).

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Heavy Ion Induced Modification of Superconductors

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The complex structure of magnetic flux lines in high- T_c superconductors and their interaction with material defects is a field of interesting basic research with some relevance also for applications. Swift heavy ions have shown to be a unique tool for the controlled introduction of correlated disorder in the form of columnar defects acting as optimum pinning sites. For this purpose GSI facilities are used by several groups from outside. These collaborations combine the advantages of heavy ion induced columnar defects of well defined density and orientation with expertise in preparation of superconducting samples (crystals, thin films, tapes) as well as special measurement techniques (transport current, magnetization, HRTEM, force displacement, ac-susceptibility, flux transformer ...). Only two of the topics that were investigated during the last year can be mentioned in this short report.

The existence and characteristics of a Bose-glass phase of vortex lines in the presence of correlated disorder as predicted by theory is of current interest. Earlier we got some evidence for a Bose-glass behavior of the vortex system in Bi-2212 epitaxial films after irradiation with very heavy ions [1]. The applicability of the theory of Bose-glass transition in the case of Bi-2212 is puzzling, taking into consideration the large intrinsic anisotropy of this compound. Possibly this can be explained through an increase of the tilt modulus in the presence of columnar defects. $YBa_2Cu_3O_{7.5}$ is known to be much less anisotropic and therefore should be favorable for the observation of a Bose-glass. Dynamic scaling analyses of the ac conductivity measured in $YBa_2Cu_3O_{7.5}$ thin films between 1 and 190 kOe before and after heavy ion irradiation parallel to the c axis reveal continuous phase transition to superconducting glassy vortex states. A first unambiguous evidence for the predicted Bose-glass behavior being stable up to the matching field of $H_\Phi = 40$ kOe is found [2]. The critical exponents differ significantly from those obtained for $H \perp c$ and those reported recently for twin-free crystals, suggesting that the inherent uncorrelated disorder in thin films reduces the vortex line wandering, and therefore enhances the Bose-glass transition.

Because of the large intrinsic anisotropy of Bi-2212 the mixed state is of quasi-2D nature and the vortices in pristine Bi-2212 samples behave as independent stacks of pancakes rather than flux lines. As already mentioned above, correlated disorder possibly increases the line tension and vortices pinned by columnar tracks in heavy ion irradiated Bi-2212 may behave as well coupled vortex lines. But no uniaxial enhancement of pinning was found in thin films from transport measurements [3], although these measurements were extended from dose equivalent fields of 2T and 1T down to 0.3T to avoid a reduction of inter-layer coupling by strong intra-layer coupling at high vortex densities.

Measurements in the flux transformer geometry are a useful means of probing the dimensionality or longitudinal correlation of vortices in the liquid phase. Possible non-local effects in the electrodynamic behavior should be directly related to the vortex dimensionality. The finite line tension of a vortex can induce a voltage distantly from the position where non-uniform currents act on it: precisely the situation which is probed by the flux transformer geometry. Fig. 1 presents [4] for an heavy ion irradiated Bi-2212 crystal the primary voltage $V_p = V_{23}(I_{14})$ and the secondary voltage $V_s = V_{67}(I_{14})$. V_s approaches and shows close correspondence with V_p in a small window of temperatures before both disappear. No such peaks in V_s were observed before irradiation. An analysis with local electrostatics of the apparent resistivities parallel (ρ_c) and perpendicular (ρ_{ab}) to the c-axis with the current injected into I_{14} and I_{18} respectively shows consistency. Therefore we suggest that the close correspondence between V_p and V_s arises, not because of non-local effects, but ρ_c becomes small resulting in an almost uniform Lorentz force.

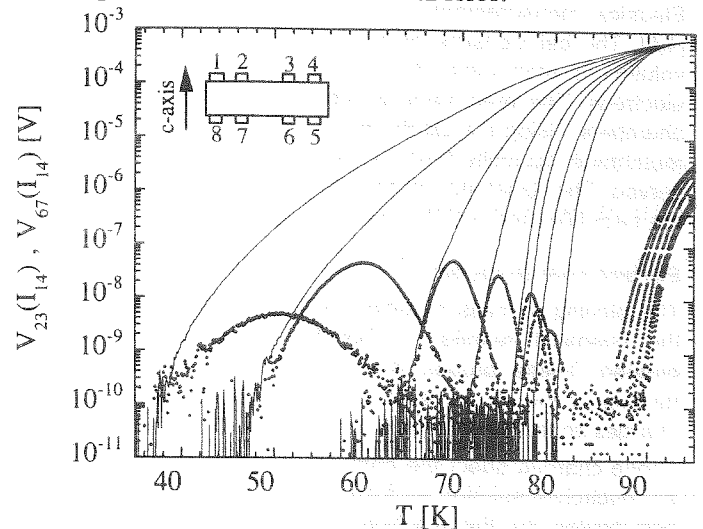


Fig. 1: Primary $V_{23}(I_{14})$ - solid line, and secondary $V_{67}(I_{14})$ - points, voltages for a Bi-2212 crystal with columnar defects // c // B of 0.1, 0.2, 0.3, 0.5, 0.7, 1.0 and 1.3T. $B_\Phi = 0.5$ T. The inset shows the contact configuration.

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