# EUROPEAN ORGANIZATION FOR NUCLEAR RESEARCH

Proposal to the ISOLDE and Neutron Time-of-Flight Committee

# PAC studies of isolated small molecules: The Pb nuclear quadrupole moments and further cases

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#### Abstract

Nuclear quadrupole moments Q offer unique information about the charge structure of nuclei. For ground and long-lived states atomic spectroscopy has by now resulted in valuable precision data for many systems. For shorter-lived excited states this method is not applicable, however.

For several particularly interesting elements precise values of the nuclear quadrupole interaction in free molecules is the best route towards accurate values for Q. In project IS640 we have demonstrated that Perturbed Angular Correlation (PAC) measurements of isolated molecules are feasible, and we have applied this to Cd and Hg nuclei. The primary purpose of the presently proposed series of PAC experiments is to obtain such data also for lead nuclei, measuring the 4<sup>+</sup> excited state of <sup>204m</sup>Pb in some simple free molecules (PbO, PbS, PbSe, PbTe), and, combined with state-of-the-art quantum chemistry calculations, obtain a reliable value for Q of this state. This result will normalize Q also for several other Pb isotopes.

To explore further the potential of the newly developed technique, pilot experiments are proposed to investigate non-linear molecules (<sup>204</sup>PbCl<sub>2</sub>), previously unknown simple cases (<sup>111</sup>CdSe), and charged molecules produced in  $\beta^{-}$  decay (<sup>117</sup>InI<sub>2</sub><sup>+</sup>).

Two cases earlier proposed in IS640, Cd-dimethyl and HgBr<sub>2</sub>, could not be measured for technical production reasons. The completion of this project is particularly important for theoretical studies of the intermolecular interaction, as the corresponding molecular solids are well investigated.

Requested shifts: 8 shifts (split into 4 runs over 2 years)

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# Motivation

All measurements of nuclear quadrupole interaction primarily determine an interaction frequency  $v_Q=eQVzz/h$ . In order to extract from this the nuclear quadrupole moment Q, the value of the electric field gradient (EFG) Vzz for the system studied must be known. This then has essentially to come from theory.

Historically atomic systems had to be employed for determining precise values of nuclear quadrupole moments. With the advent of modern quantum chemistry techniques, however, also the accuracy of EFG calculations in molecules has reached the stage of 1% precision in favourable cases [1]. Theoretical calculations for solids, on the other hand, can usually not be trusted below the 10% level, though the accuracy of the experimental interaction frequency obtained by various methods (NMR/NQR, PAC/PAD, Moessbauer spectroscopy) is generally much greater.

To date all Perturbed Angular Correlation (PAC) measurements had been performed in solids. In liquids the molecular reorientation averages out the EFG, so that at best only relaxation processes have been seen for small molecules. Very large (biological) molecules, not of interest for the present purpose, can in principle be slowed down in viscous liquids to effectively (on the PAC timescale of 100 ns) fixed state. In gases, on the other hand, molecules are freely rotating, and the effective EFG is the projection on the rotation axis. Only for linear molecules, where the rotation axis is always perpendicular to the molecular one, a unique number for Vzz results, -½ the molecular value. Intermolecular (and wall-) collisions, however, will change the rotational axis, again leading to relaxation processes due to motional averaging. To avoid these, the free flight time of the molecules must be on the PAC timescale, requiring pure vacuum conditions. For this reason early attempts of such experiments in the 1970s had to fail. With the availability of pure isotope samples at ISOLDE, however, we were recently able to overcome these technical problems. Details in [2] and [2SM].

Within project IS640 we have developed the PAC technique for the measurement of  $v_Q$  for simple linear molecules and applied it, in conjunction with state-of-the-art molecular theory, to determine Q for <sup>111</sup>Cd (5/2<sup>+</sup>) and <sup>199</sup>Hg (5/2<sup>-</sup>) to high precision [2]. This then has resulted also in precision Q for excited states in other nuclei of these elements using previous PAC/PAD results. We now intend to apply the developed method to a particularly relevant nuclear moment and test its applicability for new types of experiments.

# Primary experiment goal

The primary aim of the present proposal is to apply the same method to obtain a reliable value of Q for the 4<sup>+</sup> isomeric state (256ns) at 1274keV in <sup>204</sup>Pb. A precise value for this nuclear moment is necessary to normalize the results of various PAD studies of excited states in neutron deficient lead isotopes [3-7]. These states and their nuclear moments are the subject of modern shell model calculations, e.g. [8]. To obtain our goal we propose to determine  $v_Q$  for some simple molecular <sup>204</sup>Pb systems and to perform the associated molecular theory calculations.

## Pilot test experiments

In the present proposal we also intend to perform some pilot experiments to test the application of the newly developed technique to more exotic cases:

a) Technically also the measurement of non-linear molecules represents in principle no problem. Since the rotation axis is then not necessarily perpendicular to the EFG, however, one expects a multitude of interaction frequencies that will lead to a non-periodic PAC pattern. As an example we shall measure PbCl<sub>2</sub>, a bent molecule, where a qualitative model predicts a still useful spectrum. Theoretical work for a fully quantitative treatment is in progress.

b) The new PAC method should allow us also to investigate very high boiling compounds with limited thermal stability that have escaped spectroscopic studies to date. As an example we chose CdSe, studied at high temperature with excess Se, where the technical procedures of the Pb projects should be applicable. The result would represent a stringent test of the accuracy of the theoretical EFG result also in the case of an expected rather small value.

c) Since element-changing nuclear decay preceding the PAC cascade will in most cases lead to a disruption of the molecules, one to date is essentially limited to isomeric decay cases, <sup>111</sup>Cd, <sup>199</sup>Hg, and <sup>204</sup>Pb being by far the most favourable isotopes. In principle, however,  $\beta^{-}$  decay preceding the  $\gamma$ - $\gamma$  PAC cascade will to a very large extent produce only a singly ionized molecule. Since our technique just looks at this entity for a short time in free flight, it should produce singular spectroscopic information on ionized molecules. As a test case we plan to measure the <sup>117</sup>In  $\gamma$ - $\gamma$  cascade in the decay of <sup>117</sup>CdI<sub>2</sub>, produced as in IS640. Unfortunately, the <sup>117</sup>Cd beam from the Sn spallation target is not suitable for this PAC case, thus the low-spin <sup>117</sup>Cd isomer source has to be produced through <sup>117</sup>Ag decay.

# Experiments completing the IS640 program

One important aspect of the measurement of the EFG in free molecules, for cases where the corresponding molecular solid is known, is the possibility to test the electronic structure change in a unique way. Within IS640 we have been successful in only one of these cases, HgCl<sub>2</sub>. In preliminary calculations we were able to give a semi-quantative explanation for the measured effect [2]. Our conclusions would be much better founded if two more cases were available, as originally foreseen in the IS640 project. These two cases, HgBr<sub>2</sub> and cadmium dimethyl, could previously not be measured due to simple technical problems:

a) For HgBr<sub>2</sub> precision data for the solid are available [9], but earlier theoretical calculations for the free molecule in this case [10] predict a solid-gas shift in complete disaccord with what we expect from our HgCl<sub>2</sub> results.

b) In the case of  $Cd(CH_3)_2$  we had earlier used the measured interaction in the solid [11] to confirm the earlier suggestion [12] of an appreciable error in the generally used Q for <sup>111</sup>Cd [13]. To do this, however, we were forced to approximately estimate the (small) solid-gas shift. A direct measurement of this quantity would thus be highly valuable.

The relevant characteristics of the isotopes to be used for the now proposed measurements are summarized in table 1. For the chosen cases their relevant properties, together with the results of initial density functional (DFT) calculations, are summarized in table 2.

	T <sub>1/2</sub> (min)	γ <sub>1</sub> (keV)	γ <sub>2</sub> (keV)	t <sub>1/2</sub> (ns)	Ι	A <sub>22</sub>	Q (mb)	E <sub>rec</sub> (eV)
<sup>204m</sup> Pb	67.2	912	375	265	4+	.12	440	.85
<sup>117</sup> Cd/In	150	89	344	54	3/2+	2	-570	.05
<sup>111m</sup> Cd	48.6	151	245	84	5/2+	.1786	664	.11
<sup>199m</sup> Hg	42.6	374	158	2.45	5/2-	.251	676	.38

**Table 1**: Half-life of the isotope ( $T_{1/2}$ ), gamma-1 ( $\gamma_1$ ) and gamma-2 ( $\gamma_2$ ) energies, half-life of intermediate state ( $t_{1/2}$ ), spin (I), gamma anisotropy ( $A_{22}$ ), nuclear quadrupole moment (Q) and recoil energy ( $E_{REC}$ ).

#### **Theoretical basis**

As it has been demonstrated for several cases, including our IS640 results for Hg, modern methods of quantum chemistry are able to compute the EFG in small molecules quite precisely. We intend to apply the techniques we used earlier [2,13] within the present collaboration.

The gas phase PAC experiments are obviously performed on molecules that are vibrationally and rotationally excited at the measuring temperature. For the strictly linear molecules the rotational axis will always be perpendicular to the molecular axis, so that only the effective Vzz will possibly depend on the rotation/vibration state. Applying modern quantum chemistry methods, techniques to incorporate the small vibrational effect in the experimental analysis have been worked out in our previous experiment [2SM]. This leads to a slight spread of Vzz, resulting in a damping of the measured perturbation pattern.

For the nonlinear molecules,  $PbCl_2$  as example, a complete quantum-mechanical treatment of the coupling between rotational J and nuclear I is necessary, including the full Vxy matrix in the molecular frame. The straightforward but rather complex mathematical treatment of this system is being developed. We expect, however, only a minor modification of the standard perturbation pattern for the effectively quasi-linear molecule  $Cd(CH_3)_2$ .

A further complication in the analysis of the proposed experiments would in principle be the recoil effect of the first gamma ray in the PAC cascade. This recoil energy, see Table 1, will lead to additional vibrational excitation. It may again be estimated to lead to a small further Vzz spread. This up to now not included effect will be more important for the proposed measurement of the molecular ion  $InI_2^+$  due to the preceding beta decay.

#### Sample preparation and systems chosen

The main challenge in performing PAC measurements on isolated molecules is to produce them in a clean condition and at sufficiently small pressure that the time between intermolecular (and surface) collisions is longer than the typical PAC observation time. In Table 2 the free flight time necessary  $t_{PAC}$  and the maximum molecular density acceptable  $p_{max}$  for the cases considered is summarized together with the assumed temperature  $T_{exp}$ needed for keeping the molecules in the gas phase, rather than sticking at a container surface, as estimated for a vapour pressure of 1 hPa (.001 atm). The number of radioactive atoms needed for a PAC experiment with a source half-life of around 1 h, the present case, a representative detector solid angle of .2 steradian, and a count rate of optimally 50000 s<sup>-1</sup>, amounts to 10<sup>10</sup>. In a gas volume of 1 cm<sup>3</sup> as example for a typical sample container, this leads to a pressure of 4\*10<sup>-5</sup> Pa, well below an easily obtainable pressure of 10<sup>-2</sup> hPa and even more so below the estimated maximum allowable pressures in Table 2. Converting the maximum allowed pressures from Table 2 into matter acceptable in a sample gas volume of 1 cm<sup>3</sup> results in typically 0.5 mg amounts. In practice one would like to stay at least a factor of 10 below these values. Presumably at this relatively large amount of material the rest gas pressure due to other impurity species inevitably present will not be a severe problem.

Unfortunately, the diatomic Pb molecules considered as the only sure candidates for the moment determination have very high boiling points. The same holds for CdSe. This will require quartz containers and high-temperature ovens for the measurement. For sample production the procedure developed in IS640 for the rather volatile molecules [2SM] will thus not be suitable directly. We will modify it by implanting the radioactive ion beam directly into a very thin evaporated layer, less than 1 mg/cm<sup>2</sup>, of the appropriate compound on a small quartz slide of 1-2 mm width that can subsequently be brought into the quartz container and then simply sealed under vacuum.

**Table 2**: Properties of molecules to be used in this study: Melting point (mp), boiling point (bp), internuclear distance (R),  $v_Q$  for molecule ( $v_Q^{mol}$ ), measurement temperature ( $T_{exp}$ ), PAC time range ( $t_{PAC}$ ) required, collision time at 1 atm ( $t_{col}$ ), maximum pressure allowed ( $p_{max}$ ).

	mp	bp	R	$\nu_Q^{mol}$	$T_{exp}$	$t_{PAC}$	$t_{col}$	$p_{max}$
	[C]	[C]	[Å]	[MHz]	[C]	[ns]	[ns]	[hPa]
PbO	887	1200	1.922	1383	800	120	4.2	13
PbS	1113	1200	2.287	1319	800	120	3.1	10
PbSe	1078	1100	2.402	1234	750	120	3.1	9
PbTe	924	1000	2.595	1138	700	140	2.7	6
PbCl <sub>2</sub>	501	951	2.444	1000	700	50	3.1	20
Cd/InI <sub>2</sub>	388	744	2.582	330	550	100	3.4	10
CdSe	1240	1300	2.38	56	900	250	2.4	4
CdMe <sub>2</sub>	-3	106	2.128	1000	25	40	5.2	13
HgBr <sub>2</sub>	236	322	2.383	1410	200	15	5.3	57

For the more volatile compounds  $PbCl_2$  and  $CdI_2$  the successfully applied technique of IS640 can be employed unchanged. For HgBr<sub>2</sub> the very high volatility of this compound will require a minor change by first evaporating elemental Hg into the sample container and then sealing it with a small amount of Br<sub>2</sub> gas.

In order to synthesize cadmium-dimethyl in the minute quantities required one has to construct a special small glass apparatus where the conventionally produced  $CdI_2$ , in a minute glass boat, will be reacted with a Grignard reagent (MgBrCH<sub>3</sub>) in a high boiling solvent, presumably hexyl ether. The produced  $Cd(CH_3)_2$  will then have to be distilled in an argon stream into the final glass sample container kept at low temperature and sealed under vacuum.

### Complementary 'standard' PAC measurement

The Q for the <sup>199</sup>Hg PAC state as determined in experiment IS640 is also important for normalizing previous PAD measurements for other Hg isotopes, performed in solid mercury [14,15]. Unfortunately, the very early data for <sup>199</sup>Hg in Hg [11,16], obtained half a century ago, are not of the presently achievable precision. It is thus suggested to make a more precise determination as clearly possible with modern measuring equipment and the pure ion beam from ISOLDE. This would actually not require additional beamtime and could be performed completely in parallel with other Hg PAC experiments.

## Possible future extension: Matrix isolation (MI) experiments

The observation of simple molecules embedded in an inert matrix is a well-developed method for various spectroscopic techniques, Infrared-, Raman-, Ultraviolet-, Mössbauerand others [17]. It is therefore astonishing that no PAC experiments have yet been reported with this technique. Traditionally rare gas matrices are chosen in this field to have as little as possible interaction with the embedded molecules. This requires very low measuring temperatures, typically 20 K. Obviously in an interpretation of such data the (small) matrix effects will have to be taken into account.

Of particular interest would be a few pioneering PAC experiments using the MI technique. Clearly measurements of the linear Cd and Hg halides, where the free molecule data are now known from IS640, in frozen krypton will be prime candidates. For these an extensive series of MI experiments has already been performed using infrared spectroscopy [18]. One could thus be rather confident that unexpected difficulties will not arise. Since also nonlinear molecules could be studied with such an approach, however, many further candidates exist for other PAC isotopes as well. In the context of the present proposal, it would be highly desirable to perform such a PAC experiment for the PbCl<sub>2</sub> molecule.

To date no experimental data for the change of the EFG due to the matrix are available at all to test theoretical models. These are important for cases where the measurement of the quadrupole interaction (by Moessbauer spectroscopy) to determine Q (Sn, Fe) is only possible for matrix-isolated molecules.

At present the necessary technical facilities do not exist at ISOLDE, but a construction should be quite straightforward.

Implanted beam	Probe element	Type of experiment	Approx. Intensity [at/µC]	Target / Ion source	Required atoms per sample	n° of shifts
<sup>204m</sup> Pb	<sup>204</sup> Pb	γ-γ ΡΑϹ	10 <sup>8</sup>	UC/ RILIS	$2 \ge 10^{10}$	4
<sup>111m</sup> Cd	<sup>111</sup> Cd	γ-γ ΡΑϹ	10 <sup>9</sup>	Molten Sn/ VADIS	2 x 10 <sup>10</sup>	2
<sup>117</sup> Ag	<sup>117</sup> In	γ-γ ΡΑϹ	10 <sup>9</sup>	UC/ RILIS	2 x 10 <sup>10</sup>	1
<sup>199m</sup> Hg	<sup>199</sup> Hg	γ-γ ΡΑϹ	10 <sup>9</sup>	Molten Pb/ VADIS	2 x 10 <sup>10</sup>	1

# **Summary of requested shifts:**

# **References:**

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# Appendix

#### **DESCRIPTION OF THE PROPOSED EXPERIMENT**

The experimental setup comprises: (name the fixed-ISOLDE installations, as well as flexible elements of the experiment)

#### Part 1

Part of the Choose an item.	Availability	Design and manufacturing
Part 1: SSP-GLM Standard or Biophysics collection chamber (ISOLDE hall)	Existing	☐ To be used without any modification
Part 2: Chemical Laboratory (508/R-002)	Existing	To be used without any modification
		To be modified
	New	Standard equipment supplied by a manufacturer
		CERN/collaboration responsible for the design and/or
		manufacturing
Part 3: Experimental PAC zone (508/R-008)	Existing	Various PAC spectrometers
•		To be modified
	New	Standard equipment supplied by a manufacturer
		CERN/collaboration responsible for the design and/or
		manufacturing
[insert lines if needed]		

#### HAZARDS GENERATED BY THE EXPERIMENT

*(if using fixed installation)* Hazards named in the document relevant for the fixed [COLLAPS, CRIS, ISOLTRAP, MINIBALL + only CD, MINIBALL + T-REX, NICOLE, SSP-GLM chamber, SSP-GHM chamber, or WITCH] installation.

Additional hazards:

Hazards	Part 1	Part 2	Part 3				
Thermodynamic and fluidic							
Pressure							
Vacuum	.00000001 - 1 Bar], .1l	.00000001 - 1 Bar], .1l	.00000001 - 1 Bar], .1l				
Temperature	273 К	273-700 К	77-1000 K				
Heat transfer							
Thermal properties of materials							
Cryogenic fluid	Liquid nitrogen for SSP-GLM Biophysics collection chamber (201) and sample holder cooling (0.11)		Liquid nitrogen (11)				
Electrical and electromag	netic						
Electricity	230 V, max 10 A single phase	230 V, max 10 A single phase	230 V, max 10 A single phase				
Static electricity							
Magnetic field	[magnetic field] [T]						
Batteries							
Capacitors							
Ionizing radiation							
Target material	Frozen water, evaporated samples						

Beam particle type (e, p, ions,	204Pb, 111Cd, 117Ag, 199Hg		
etc)			
Beam intensity			
Beam energy	60 keV		
Cooling liquids	Liquid nitrogen		Liquid nitrogen
Gases	[gas]	Ar, ArH2, ArBr, He	
Calibration sources:			
Open source			
Sealed source	[ISO standard]		
Isotope			
Activity			
Use of activated material:		Chamistry proparation as	Discod in DAC spectrometer
Description	Described in the proposal	described in proposal text	for measurement
	Removal from chamber	uescribed in proposal text	for measurement
	transport to chemical		
	laboratory in standard Pb		
	shielded container		
Dose rate on contact	Max 0.3 micro Sievert /h	Max 0.3 micro Sievert /h	Max 0.3 micro Sievert /h
and in 10 cm distance			
• Isotope	204mPb, 111mCd, 117Cd,	204mPb, 111mCd, 117Cd,	204mPb, 111mCd, 117Cd,
	199mHg	199mHg	199mHg
Activity	Max 5 MBq per sample	Max 5 MBq per sample	Max 5 MBq per sample
Non-ionizing radiation			
Laser			
UV light			
Microwaves (300MHz-30			
GHz)			
Radiofrequency (1-300MHz)			
Chemical			1
Toxic	[chemical agent], [quantity]	PbCl2, Cdl2, HgNO3 0.5 mg	
		Cd-dimethyl 0.1 mg	
Harmful	[chomical agent] [quantitu]	PbO, PbS, PbSe, PbTe 0.05mg	
	[chemical agent], [quantity]		
and substances toxic to	[chemical agent], [quantity]		
reproduction)			
Corrosive	[chemical agent], [quantity]		
Irritant	[chemical agent], [quantity]		
Flammable	[chemical agent], [quantity]		
Oxidizing	[chemical agent], [quantity]		
Explosiveness	[chemical agent], [quantity]		
Asphyxiant	[chemical agent], [quantity]		
Dangerous for the	[chemical agent], [quantity]	Pb, Cd, Hg chemicals	
environment			
Mechanical	1	1	1
Physical impact or	[location]		
mechanical energy (moving			
parts)	[lesetion]		
(Sharp, rough, clippony)	[location]		
Vibration	[location]		
Vehicles and Means of	[location]		
Transport	Freedoment)		
Noise	<u>،</u>	<u>،</u>	
Frequency	[frequency] [Hz]		
Intensity	[		
Dhysical	1	1	1

Confined spaces	[location]		
High workplaces	[location]		
Access to high workplaces	[location]		
Obstructions in passageways	[location]		
Manual handling	SSP protocol	ISOLDE Chemistry Laboratory	SSP protocol
		protocol and material safety	
		data sheets	
Poor ergonomics	[location]		

#### 0.1 Hazard identification

3.2 Average electrical power requirements (excluding fixed ISOLDE-installation mentioned above): (make a rough estimate of the total power consumption of the additional equipment used in the experiment)