COLUMBIA UNIVERSITY, DIVISION OF WAR RESEARCH

Project DSM

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July 3, 1943.

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INTRODUCTION

A short history of our project may be of value. The idea of utilizing photochemistry to effect a separation of the isotopes was explored here at Columbia University with gases. In Berkeley, two investigators, Lipkin and Weissman, examined the photochemistry of a number of compounds in the solid state and in solutions. Their work followed a suggestion of Pringsheim that, since the intensity and half-lives of the fluorescence spectra of uranyl salts indicated the fluorescence to be forbidden radiation, some lines of the spectra may have been induced by the nuclear magnetic moment of the isotope 235; i. e., that a portion of the spectrum had arisen from the odd isotope and not from the even isotope, and if this radiation is absorbed and produces a chemical reaction, the reacting molecules would be relatively richer in 235 than in 238. A number of such instances were well known. Both the project at Columbia University and in Berkeley were badly misled by errors in the analyses of isotopic ratios and much of their time was wasted. The analytical technique has since achieved high precision and reliability.

Only the most general and qualitative considerations had entered into the photochemical experiments. The project we are considering today has a different emphasis. It is a thoroughgoing investigation to discover prior to any program of photochemical separation what the spectra of various uranium compounds represent so as to detect the lines of the spectra, which are preferentially due to 235 rather than 238. It is to be expected that the interactions which distinguish one isotope from another can produce a change in but a small portion of the spectrum. The situation is then most heavily loaded against any "pot shots" and the possibility of success depends sharply on understanding the microscopic origin of the spectra and on photochemical reactions which the properly selected radiation may induce.

Only sharp spectra can be regarded as suitable, both from the point of view of interpretation and for discriminating and utilizing the small difference in the spectra which would distinguish the isotopes unless some windfall should occur. For this reason the sharpening of spectra which low temperatures bring about is important.

A chief contribution of the previous work was the discovery that there is nothing especially rare about systems in which

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uranium reacts photochemically at the lowest temperature tried - that of liquid nitrogen. (Appendix 3)

The construction of the laboratory we are now working in was begun last February, and a considerable part of the effort and time has gone into more construction. The responsibility for spectroscopy was given over to Duncan; for the photochemistry, to Merrick; and for chemical preparations and analyses, to Gilbertson. The interpretation was put chiefly into the hands of Maria Mayer. Teller was added as consultant toward the end of February. About six weeks ago I began work here when liquid hydrogen became available. I had been stressing, and others were also of the opinion, that the refinement achieved in the spectra at the temperature of liquid hydrogen was needed for getting within reasonable time at that selection of spectra and substances we required for photochemical separation. In passing, I may say that the width of lines in the spectrum of uranyl nitrate had been measured (Joos and Duhm, Ann. Phys. p. 123 (1936)) and found to be 5 A at the temperature of liquid air and 1 A at the temperature of liquid hydrogen. The spectra of other uranyl compounds are considerably sharper, at least at higher temperatures, than that of the nitrate. Once the lines of the spectra chiefly due to 235 are determined at the temperature of liquid hydrogen, they may be followed to higher temperatures. It has always been the intention to carry out the photochemistry at higher temperature, say at the temperature of liquid air. The necessary precautions for handling liquid hydrogen in the laboratory have now been completed and last week our first spectra were taken at that temperature.

SCIENTIFIC BASES OF PHOTOCHEMICAL SEPARATION

There are principally two properties which may be used to distinguish the two isotopes spectroscopically: the difference in the masses of their nuclei and the difference in their spins. The difference in the masses would be expected to introduce about 1 cm^{-1} separation in one of the fundamental vibrational frequencies of uranyl ion and also of the hexafluoride and proportionally greater separations in the harmonics. As regards spin, we may be confident that the nucleus 235 (see Appendix 5) has a spin and that the nucleus 238 definitely has none. The spin of 235 may manifest itself in the spectra in several ways: one way as hyperfine structure, and another, of more importance for this project, in the appearance of spectral lines induced by the

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nuclear magnetic moment, which do not appear in the absence of such moment, i.e., with the isotope 238. The line within the band system of the spectrum so induced may well be far enough from other lines as to offer no difficulty of resolution. For example, the line $3P_0 - {}^1S_0$ in the mercury spectrum, which arises from the odd but not from the even isotopes, is seventeen hundred wavenumbers from the nearest lines of the spectrum.

SUCCESSFUL PHOTOCHEMICAL SEPARATION OF OTHER ISOTOPES

That photochemical methods have been successful in separating isotopes is well known. In mercury Mrosowski (Zt. f. Physik 78, 826 (1932)) had established that some of the lines originate in the odd isotopes and, based on these lines, he suggested a photochemical method for obtaining a separation. Zuber (Helv. Acta Phys. 9, 285 (1936)) employing this method achieved a four-fold enrichment. Recently, Kuhn, Martin, Mldan (Z. Physik Chem. B 50, 213 (1941)) effected a separation of the chlorine isotopes in ClO_2 ~~and a remarkably successful separation in the hydrogen isotopes was obtained by Farkas and Farkas (Trans. Faraday Soc. 34, 1120 (1938)) by irradiating solutions of sodium bisulfite in water. The heavy hydrogen isotope was enriched eighteen fold; the mechanism for this reaction is still obscure. The extremely high value was confirmed by Crist at Columbia University~~.

POSSIBILITIES OF SUCH SEPARATION IN URANIUM METHODS OF PRESENT PROJECT

Because of the low vapor pressure of elementary metallic uranium, even at high temperatures, it seems impracticable to attempt photochemical fractionation of atomic uranium. The more volatile compounds of uranium are rather complex and in the gaseous state give rise to diffuse spectra with little resolved structure. Only a few of these have been examined, notably the hexafluoride, for which some of the vibrational frequencies are known from Raman data and also from infra-red data. Steps have been taken here to take the absorption spectrum of the hexafluoride under considerably more favorable conditions than formerly.

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On the other hand, the spectra of many uranium compounds in the solid state and in some solutions are almost unique in their sharpness, comparing favorably with the rare earths. This property is well known as regards the absorption and fluorescence spectra of the uranyl salts. It holds also for the absorption spectra of salts of uranium in the quadrivalent and trivalent states (F. Ephraim & M. Mezener, Helv.Chim.Act. 16, 1360 (1933)). Both the fluorescence and absorption spectra of the uranyl salts consist of rather sharp lines clustered in groups which repeat their pattern at constant intervals. The intervals differ slightly from salt to salt, while the pattern of the groups reflect the symmetry of the environment of the uranyl ion. The present interpretation accounts for this sharpness by postulating an excited quantum state which has an electron in an inner f orbit, shielded from perturbations by the outer completed electronic shells.

A beginning has already been made in the investigation of these compounds with particular reference to those crystals where X-ray data (Zeitschrift Z. Kryst 94, 312 (1936)) have furnished information on the configuration of atoms about uranium, sodium uranyl acetate, rubidium uranyl acetate. There is good reason to believe that sharp spectra will be furnished by some solutions of uranyl salts in organic solvents which are mobile just above the temperature of liquid air. Uranyl acetate in a mixture of isopentanes, ether, and alcohol was found by Lipkin and Weissman to give especially sharp structure at the temperature of liquid air.

Our program is directed at understanding the spectra of uranium compounds so as to identify the lines which distinguish the U235 isotope. For identifying these lines a complete analysis of the spectra is unnecessary. However, enough should be understood so that it would lead to a selection of those substances which have the greater differences in the spectra of the isotopes. After the important lines are found they will be used in photochemical separations. Already photochemical experiments are being carried out to discover which substances react photochemically.

To single out the part of the spectrum due to the odd isotope, the action of a magnetic field on the spectrum of the chosen substance will be of great assistance. The external magnetic field effects the spectra of all the uranium in the same way but to a greater degree than the nuclear magnetic moment affects the spectrum of the odd isotope. The equivalent field of the nuclear moment is estimated (appendix) at from 2000 to 10,000 gauss. External fields of 20,000 gauss are available. The intensity of the radiation forced by the field is quadratic in the field strengths and the effect of the external field is on all the uranium present and not only on a small fraction.

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A direct method for detecting lines of the spectrum peculiar to the isotope 235 is to make use of samples enriched in the isotope. Such samples are now obtainable. Lipkin and Weissman just before being transferred to another project had taken some spectra of material containing 16% U235. One of the lines in rubidium uranyl nitrate was found intensified five-fold and several lines in sodium uranyl acetate about fifty per cent. Such figures lead to a ratio in the transition probabilities A235/A238 of 26 and 4.3, respectively, which seemed unreasonably high. However, they later found that the intensification of the line in rubidium uranyl nitrate was due to a hydrate mixed with the anhydrous crystals.

It may be worthwhile at this point to mention that if one absorption line in a band of this enriched sample was only 2% more intense than the corresponding line from ordinary uranium material, photochemical separation would be almost ready for the engineer and big scale production. (appendix table 1). This corresponds to a ratio of 1.12 in the absorption coefficients of the isotopes for that line.

Careful work can avoid the presence of mixtures so that comparisons of the spectra, especially the absorption spectra, would be trustworthy. The work with the magnetic field requires no such care since the field works on the identical sample whose spectra had been taken without field.

The program for investigating the spectra to discover the most favorable substances includes many compounds of uranium of different valences with the uranium atoms in different environments of known configuration and symmetry wherever possible. The spectra of the crystals are to be simplified by examining polarization spectra of uniaxial crystals. Also, the spectra are to be analyzed into component spectra according to their multipole character. This analysis consists of a comparison of the polarized spectra examined with light coming from crystals at different directions relative to the crystal axis. Such observations permitted Deutschbein (Ann.der Physik 36, 133 (1939)) to determine that the radiation from a crystal of a rare earth consisted of two species: one of electric dipole radiation and the other magnetic dipole radiation. His results were confirmed by wide-angle interference measurements from solutions of the same rare earth (S. Freed and S.I. Weissman Phys. Rev. 60, 440 (1941)). The use of crystals rather than solutions will permit us to pick up such differences in the nature of the

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radiation at the lowest temperatures where the disturbances from random electric fields are small and it will then be relatively easy to continue to identify the radiation at higher temperatures and in solutions.

So far, we have found no clear-cut method of estimating the relative importance of random electric fields and the nuclear magnetic field in forcing forbidden radiation. The intensity of the lines forced by the nuclear field is computed in an appendix 1. The fact that fluorescence from uranyl ions has a half life of 10^{-4} second, even in solutions at room temperature, where the random fields are considerable indicates that in many compounds these fields are not particularly strong. It seems probable that a judicious choice of substances having fields of high symmetry and weak fields about the uranium atoms would select those instances where the competition from random fields is not serious. We shall see that only a small percentage effect of the nuclear moment is sufficient. It has been found in some of our work in Chicago that the local symmetry of the microscopic fields about ions in some solutions are higher than in many crystals.

The investigation will be extended to the conditions under which the photochemistry would apply on a practical scale, that is, to clear solutions at the temperature of liquid air. From experience with the rare earths it seems likely that sharp spectra will be found in solutions at low temperatures also. Already Lipkin and Weissman have reported lines within one angstrom unit in the spectrum of uranyl acetate in organic solvents. In all probability solutions may be found whose spectra are sharper than that of the acetate.

As to effects in the spectra due to the difference in the masses of the isotopes - they are not excluded for purposes of photochemical separation even though isotopic frequency differences are not resolved in the spectrum. If photochemical excitation takes place on the high frequency side of the profile of the absorption line, an enrichment may occur since, as is shown in detail in the appendix 1, a ratio in absorption coefficients of the isotopes as low as 1.05 becomes practical and a ratio 1.10 is definitely on the favorable side.

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ESTIMATES OF COST IN LARGE SCALE PRODUCTION

A computation has been made on the following rather reasonable basis which is more fully and precisely given in the Appendix 1.

The absorption spectrum of a uranyl compound contains a photochemically useful line in one of the bands whose intensity is not less than one ten-thousandth of that of the band. Arcs may be obtained which can convert one thousandth of their electrical input into this photochemical frequency. (This ratio appears rather optimistic, but it has been surpassed in sources of resonance radiation.)

A kilogram of U_{235} ninety per cent pure can be made at the following estimated costs:

Ratio of Absorption Coefficients	Price Electrical Energy per kg. U_{235}	Power to produce 1 kg. 90% U_{235} per day Kilowatt	Cost of installation in million $
2.0	154	3.2×10^3	.24
1.5	616	1.3×10^4	.96
1.2	3360	8.0×10^4	6.05
1.1	1.54×10^4	3.2×10^5	24.1
1.05	6.16×10^4	1.3×10^6	96

It appears then that if a line is found not altogether too weak whose ratio in the absorption coefficients is more than 1.05, it may be used for producing large quantities of the odd isotope. If liquid hydrogen had to be used, a higher minimum ratio in the absorption coefficients would be necessary. However, we do not look upon such a possibility as likely. A ratio of 1.10 is already attractive and still higher very favorable.

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TIME SCHEDULE

It should be kept in mind that nothing but preliminary work has been done so far. The basic ideas are certainly correct. They have been experimentally verified, for example, by Zuber (Helv. Act. Phys. 9, 283 (1936)) who obtained a four-fold enrichment of the mercury isotopes. The present project would be content with one fortieth of that, i.e., 10 per cent. We estimate that in six months it is possible to test the applicability of the ideas to our systems. On the other hand, the results may be negative but definite, and we may be able to state with assurance that this method with powerful possibilities is not successful in the hands of the enemy. If the work does not continue, it is very probable we shall turn to this possibility again and again and at a later time be forced into such an investigation to settle the possibilities once and for all.

In conclusion, I shall quote an opinion expre ed by Teller. He said that if this approach had been taken up when the other methods had been started, this one might well have overtaken the others by this time.

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Appendix I.

Estimate of the Cost of the Photochemical Process

In order to obtain a clearer understanding of the magnitude of an effect which could be used in practice, some of the expenditures in power and money have been calculated.

Let us assume that a photochemical process has been found with a quantum yield of one, for which the ratio of A_5, the absorption coefficient of 235, to A_8, that of 238, is

$$(1) \qquad \frac{A_5}{A_8} = \alpha > 1.$$

$\alpha - 1$ is then the fractional enhancement of 235 in the material that undergoes the photochemical reaction.

To estimate the amount of light required, a cascade of reaction steps has to be built, in which the reaction products of each step are sent on to the next higher, while the remaining material is fed back to the next lower step. In each step, a little less than 1/2 of the material is advanced.

The calculations for such a cascade are identical with those for a diffusion process, and the results can be obtained directly by using the formulae developed previously*. The quantity α, as defined by (1), corresponds to the simple process factor α used by Cohen. The most efficient cascade is obtained, if the two streams entering each stage, the one from above and the one from below, have the same isotopic composition. Cohen's formulae hold for $\alpha - 1 < 1$ only. For $\alpha = 1.5$ and $\alpha = 2$ the energies obtained with these formulae are slightly too low (about 10% for $\alpha = 1.5$ and possibly by 1/2 at $\alpha = 2$).

The number of stages to obtain material containing 90% U_{235} is given by

$$(2) \qquad N = \frac{7.14}{(\alpha-1)\ \ln 2} = \frac{10.3}{\alpha - 1}$$

These values are listed in column 2 of Table I.

* Karl Cohen, A96, Total Areas, Pumping Speeds and Stages for Diffusion Apparatus.

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The number of mols of light quanta necessary to obtain 1 kg. of 90% U_{235} is

$$(3) \qquad \frac{1000}{235} \; \frac{130}{((\alpha-1)\ln 2)^6} = \frac{1161}{(\alpha-1)^2}$$

These values are listed in column 3.

It was further assumed that an arc or spark may be found which converts one thousandth of its energy input into a line of the desired frequency. This figure may be somewhat optimistic. But if it were possible to use a resonnance line, one could do a great deal better. The line needed was assumed to have a frequency of about 20,000 cm^{-1}. One mol of quanta corresponds to 57.2 kilocalories or .067 kilowatt hours; 67 kilowatt hours are needed in the arc to produce this amount of useful light. The energy required to obtain 1 kg. of the desired material is then

(4) Energy = no. of mol quanta x 67 kilowatt hours.

These numbers are listed in column 4.

The cheapest electric rates are about .2 cents per kilowatt hour. On this basis, the figures in column 5 are obtained.

The power needed in a plant which produces 1 kg. a day is given in column 6.

The price of electrical equipment alone to construct such a plant is, according to a private communication to Karl Cohen by Rhea of G. E., $75 per kilowatt. The price of this part of the installation is contained in column 7.

If the plant is to reach equilibrium in 100 days, the time which the material may spend in each step is

$$(5) \qquad t = \frac{100\,((\alpha-1)\ln 2)^2}{10.3} \text{ days} = 4 \times 10^5\,(\alpha-1)^2 \text{ seconds.}$$

These times are listed in column 8.

The time which the material must spend under the influence of the light source in order that each molecule has a chance of about 1/2 to absorb light, is, for $\alpha-1 > .05$, a fraction of the allowed holdup time listed in column 8. It may be computed in the following manner.

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It should be possible to arrange the equipment such that the total intensity is 100 watts per cm^2 (10 kilowatt over 100 cm^2 of area). Of this, only 1/1000 is light of the desired frequency. If the line in question has a frequency of 20,000 cm^{-1}, this corresponds to 2.5×10^{17} quanta per sec. per cm^2.

If σ denotes the cross section of the molecule for absorption of light of this frequency, the photochemical time is obtained from the relation

no. of quanta per cm^2 per sec. $\times \sigma \times t = 1/2$,

$$(6) \quad t = \frac{1}{\sigma\, 2 \times 2.5 \times 10^{17}} = \frac{2 \times 10^{-18}}{\sigma} \text{ sec.}$$

The cross section may be estimated from the lifetime of the fluorescent state. If this lifetime is taken as $\tau = 10^{-6}$, it follows $\sigma = 1.15 \times 10^{-16}$ cm^2. This, however, is the cross section corresponding to the total intensity of the band system. If a line were used which contains only 10^{-4} of this total intensity, the effective cross section is smaller by that factor, and one finds

(7) $t = 173$ secs.

Comparison of this with the figures in column 8 shows that in those cases which could possibly be managed (i.e., $a-1 \geqslant .05$) the intrinsic holdup is insignificant.

The ninth column contains the amount of liquid nitrogen needed; the tenth, its price at 10 cents a liter.

These figures are estimates on the pessimistic side. It is assumed that all the light energy absorbed by the molecules has to be removed by liquid nitrogen. If the chemical reaction is endothermic, this would not be the case.

The figures in column 9 and 10 are computed on the basis that the heat of vaporization of 1 liter of liquid N_2 is 47.8 kilocalories. Column 3 gives the number of mol quanta absorbed in the cascade; each mol of quanta corresponds to 57.2 kilocalories.

If the separation of the reaction products from the bulk of the material can be done at high temperatures only, more liquid nitrogen is needed to cool the material for each step. This amount, however, is small compared to the heating due to light.

If liquid hydrogen would have to be used, matters would become rather unfavorable. Due to its low density, the heat of vaporization of one liter of liquid hydrogen is only 7.5 kilocalories. The amounts needed are given in the last column.

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T A B L E I

I	II	III	IV	V	VI	VII	VIII	IX*	X	XI*
	No. of st gas to obtain 90% U_{235}	The production of 1 kg. of 90% U_{235} requires			The plant to produce 1 kg. of 90% U_{235} per day		Transit time for 100 days equilibrium	The total cooling for the production of 1 kg. of 90% U_{235} requires either		or possibly
		Mol uanta	Kilowatt Hours	Costs $	requires a power of Kilowatt	costs in electrical equipment Million $	Sec.	Liter of liquid N_2	Price of liquid N_2 $	Liters of liquid H_2
1	11	1150	7.71×10^4	154	3.21×10^3	.241	4×10^5	1382	138	8.7×10^3
.5	18	4.6×10^3	3.08×10^5	616	1.28×10^4	.96	10^5	5.53×10^3	553	3.48×10^4
.2	57	2.88×10^4	1.93×10^6	3360	8.04×10^4	6.03	1.6×10^4	3.46×10^4	3.46×10^3	2.18×10^5
.1	110	1.15×10^5	7.71×10^6	1.54×10^4	3.21×10^5	24.1	4.03×10^3	1.38×10^5	1.38×10^4	8.7×10^5
.05	210	4.6×10^5	3.08×10^7	6.16×10^4	1.28×10^6	96	1000	5.53×10^5	5.53×10^4	3.48×10^6
.02	520	2.88×10^6	1.93×10^8	3.36×10^5	8.04×10^6	6.03×10^2	160	3.46×10^6	3.46×10^5	2.18×10^7
.01	1000	1.15×10^7	7.71×10^8	1.54×10^6	3.21×10^7	2.41×10^3	40	1.38×10^7	1.38×10^6	8.7×10^7

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* If the photochemical reaction is endothermic, these magnitudes would be reduced.

Appendices

2. -

Intensification of a line occurs, in the language of perturbation theory, due to the fact that the perturbing nuclear magnetic moment mixes an allowed level with the forbidden one. The extent of the mixing is

$$\left| \frac{U_{am}}{E_a - E_m} \right|^2 ,$$

where U_{am} is the matrix element of the perturbing potential between the excited forbidden and an excited allowed level of the molecule. U_{am} has the magnitude of hyperfine structure, splitting about .5 cm^{-1}. The intensification due to the spin is then

$$\left| \frac{.5}{\nu_{am}} \right|^2 \cdot f_{ao},$$

where f_{ao} is the absorption coefficient of the "allowed" level, ν_{am} the difference in energy between "forbidden" and "allowed" excited level. The forbidden line may steal its intensity from a close lying allowed line. If ν_{am} were about 10 cm^{-1}, the weak line would receive an intensification due to nuclear spin of 2.5 x 10^{-3} of the strong line. Or it may steal its intensity from a line of electron number 1 far away, say ν= 10,000 cm^{-1}, in which case its intensity may be 2.5 x 10^{-9} electron numbers (compared to an electron number of 10^{-6} to 10^{-5} computed from the lifetime of the fluorescent state for the sum of the intensities of all lines of the band system).

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Appendices

3.

Uranyl Compound	Reducing Agent	Solvent	Temp.	Fluorescence	Reduction
Sulfite	H_2SO_3	H_2SO_4	193	Weak	Yes
Sulfite	H_2SO_3	H_2SO_4	293	None	Yes
Bromide	EtOH	EPA*	90	Yes	Yes
Phosphate	$(CH_2OH)_2$	H_3PO_4	193	Weak	Yes
Acetate	Lactic acid	Lactic acid	193	Yes	Yes
Crystalline potassium uranyl ferrocyanide	None	None	90(?)		Yes
Uranium hexafluoride	$CFCl_2-CF_2Cl$		90		Yes
	$CHF_2 - CFCl_2$		90		Yes
	$CF_2Br \quad CF_2Br$		90		Yes
	$CF_2Cl - CF_2CF_2CF_2Cl$		90		Yes
	$CF_2Cl \; CFCl \; CFCl \; CF_2Cl$		90		Yes

* Mixture of ether ethanol and isopentane.

4.

The accuracy in the analyses of the ratio of the concentration of the isotopes is within 0.1 to 0.2% and the precision of this measurement is ± 0.04%.

5.

Of the many isotopes of even atomic weight and even atomic number, all have zero spin. Of the isotopes of odd atomic weight and even atomic number, all have half integral spins from 1/2 to 9/2. There are 21 of these isotopes.

6.

Hyperfine structure splittings are of the order of 0.5 cm^{-1}. A magnetic field of 10,000 gauss gives a Zeeman effect of this magnitude.

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