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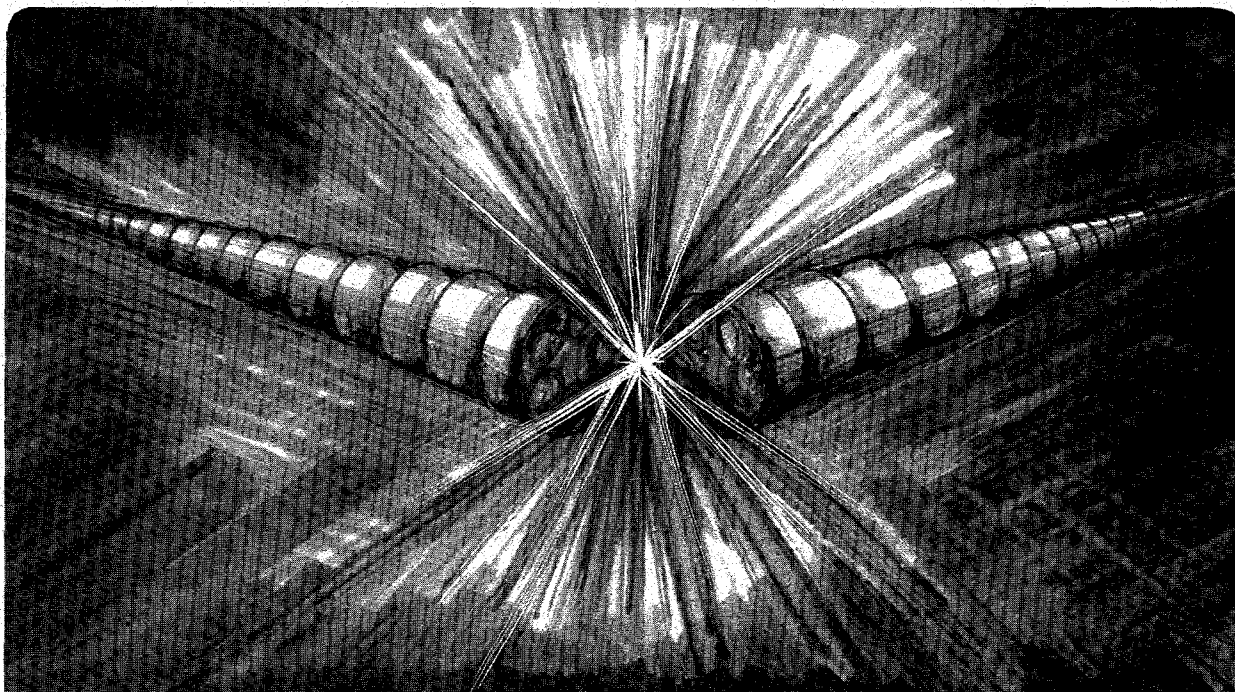
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### First Results from the High-Brightness X-Ray Spectroscopy Beamline 9.3.1 at ALS

W. Ng, G. Jones, and R.C.C. Perera

October 1995



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**FIRST RESULTS FROM THE HIGH-BRIGHTNESS X-RAY SPECTROSCOPY  
BEAMLINE 9.3.1 AT ALS\***

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**First Results from the High-Brightness  
X-Ray Spectroscopy Beamline 9.3.1 at ALS**

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**Abstract**

Beamline 9.3.1 at the Advanced Light Source (ALS) is a windowless beamline, covering the 1-6 keV photon-energy range. This beamline is designed to achieve the goal of high brightness at the sample for use in the X-ray Atomic and Molecular Spectroscopy (XAMS) science, surface and interface science, biology, and x-ray optical development programs at ALS. X-ray absorption and time of flight photoemission measurements in 2 - 5 keV photon energy along with the flux, resolution, spot size and stability of the beamline will be discussed. Prospects for future XAMS measurements will also be presented.

**1. Introduction**

Third-generation synchrotron radiation sources like the Advanced Light Source (ALS) in Lawrence Berkeley National Laboratory provide a unique opportunity for advancements in x-ray spectroscopy of atoms, molecules, and solids, because of the high brightness available[1]. Beamline 9.3.1 at the ALS is a windowless beamline, covering the 1-6 keV photon-energy range. The beamline is designed to achieve the goals of high flux and preservation of the high brightness from ALS. The anticipated beam size is about 0.4 mm in diameter, and the

estimated flux at the focus is expected to be  $10^{11}$  ph/s in 0.5 eV bandpass. The beamline was commissioned in the spring of 1995, with a prototype M1 mirror but without the M2 mirror. We will discuss the initial operation of the beamline in this configuration.

## 2. Beamline and Monochromator Design

The first mirror (M1) in beamline 9.3.1 at the ALS is located 11.75 m from the source; it collimates the beam vertically and horizontally from bend magnet 9.3. The second mirror (M2), located 15.50 m from the source, focuses the beam vertically and horizontally onto the sample at 27.25m from the source. The two-crystal monochromator is located 13.63m from the source. This configuration allows for high resolution and high brightness by passing a collimated beam through the monochromator, and by focusing the ALS source on the sample with unit magnification. The maximum horizontal acceptance of BL 9.3.1 is 8 mrad, but smaller horizontal acceptance of bend magnet radiation can be used to minimize the dominant mirror (spherical) aberrations. The sagittal and tangential radii of both toroidal mirrors (M1 and M2) are the same. The sagittal (minor) radius is 0.2585 m and the tangential (major) radius is adjustable from 1000 m to  $\infty$  (flat) in increments of 1% by elastically bending the mirror. The nominal tangential radius of the mirror is 2136 m when the mirror is positioned such that the glancing incidence angle for the principle ray is 11 mrad. A detail study of the optical design of this beamline is presented elsewhere[2,3]. Both M1 and M2 are mounted rigidly to the vacuum tank. The mirror and the vacuum tank are mounted on a motorized six struts HEXAPOD system[4]. Each strut in the system uses a stepping motor in conjunction with a linear scale closed-loop feedback system. All six struts are controlled by a window-based software system. A very attractive aspect of the HEXAPOD system is its ability to define the origin of its coordinate system anywhere within its range of travel. In our case, the origin was defined as the center of the mirror surface. The designed range of travel, rotation and resolution of the M1 and M2 mirror chambers mounted on the HEXAPOD system are shown in Table 1.

The design of our double crystal monochromator[5] is based on the original "Cowan type" monochromator[6,7], but several significant enhancements have been introduced. The primary change is the removal of all bearings from the mechanism, particularly the crossed-roller bearings used in the original design. Movement of the crystal mounts along the boomerang arms as well as all other linear motions use dovetail slides made of aluminum/bronze sliding on bases made of 304 stainless steel, with both surfaces treated chemically with a vacuum-compatible lubricant. This combination of materials is known not to seize, even in ultra-high vacuum (UHV), and the hardness of the materials permits high-precision repetitive motion. Other design modifications include a significant reduction in the number of parts and use of more robust components. Reduction in the number of parts was accomplished by eliminating the crossed-roller bearings, by eliminating unneeded adjustments in the mechanism, and by consolidating multiple pieces into integrally machined components wherever possible. Robustness was achieved simply by making many parts heavier or larger or using ribs for added strength in some larger components. Ease of alignment also has been enhanced by liberal use of fiducial marks throughout the mechanism, its support base, and the monochromator vacuum chamber. Photon-energy scanning is accomplished from outside the vacuum by a single linear feed-through coupled to a stepping motor operating in a micro-stepping mode, with 15 cm of total movement. The angle of the first crystal is monitored by a high vacuum compatible tilt sensor[8]. The resolution of the sensor is 2  $\mu$ rad with a total range of 70°. The response time of the tilt sensors are in the minute range, which is not suitable for closed loop operation but very useful as an absolute reference. The monochromator is controlled by a Labview-based data acquisition program. The alignment of the second crystal can be adjusted in-situ in both  $\theta$  and  $\chi$ . The motions are actuated by two vacuum compatible pico-motors[8], which have a range of 1.3 cm and a resolution of 30 nm. A closed loop feedback system designed to keep the final focus to within a fraction of the beam size will be installed. The system uses a 4-sector photodiode and a system of differential and summing amplifiers to feed back into the pico-motor. The monochromator operates at a based vacuum of  $2 \times 10^{-9}$  torr. The vacuum in the M1 and M2 chambers is maintained in  $2 \times 10^{-10}$  torr with the implementation of a thin carbon foil vacuum barrier

between the monochromator tank and M1 and M2 mirror tank, which allows a fast turn-around time for crystal set change.

### 3. Experimental and Initial Results

The mechanical components of beamline 9.3.1 were commissioned in the spring of 1995. A prototype M1 mirror with a fixed radius was installed in April 1995. The M2 mirror was not installed, and it is expected to be delivered in the winter of 1995. The experiments reported here were performed with the beamline in this unusual configuration. The SR source is collimated by the M1 mirror and monochromatized by the monochromator. The exit beam is collimated and it is about 3 cm x 1 cm. This unfocused beam needs to be apertured to be usable for most experiments; therefore the flux delivered to a small sample area is greatly reduced. Initial operation of the monochromator were performed with Si(111) crystal set. The first set of experiments was done with a gas absorption cell in order to check the energy calibration and resolution of the monochromator. Ion current of the sample gas is collected by a two-plate gas cell. Fig. 1 shows an absorption spectrum for Ar K edge (5 torr). This spectrum is not normalized and the feed back loop was not used. This data demonstrates the resolution and stability of the monochromator mechanism. Fig.2 is an absorption spectrum of the Cl K edge of 4 torr of  $\text{CF}_3\text{Cl}$ . This result agrees very well with previously published data[10], and the estimated energy resolution in this spectrum is 250 meV. Fig. 3 is the S K absorption spectra  $\text{SF}_6$  (5 torr). The scale for the upper spectrum has been increased 8.5 times to show the smaller structures that were not visible in the lower spectrum. This data is also consistent with published results with lower energy resolution[11].

The first time of flight (TOF) experiment on BL 9.3.1 was obtained during ALS two-bunch operation in August 1995. Since the exit beam of 9.3.1 is not focused at this time, the beam was apertured to 1 mm x 1 mm for this experiment. The flux delivered to the gas jet is only a fraction of the expected flux in a focused beam. Even in this configuration we were able to obtain TOF spectra with a reasonable count rate. Fig 4 shows a spectrum taken at a photon energy above the argon K-shell threshold (3205 eV). The data were collected using a space-focused ion TOF mass spectrometer. Because of the double-bunch mode of operation, two spectra are shown in Fig. 4, indicated by labels above or below the baseline, respectively. The time axis is scaled to give



the correct flight time for the peaks with labels below the spectrum. All of the peaks in the second spectrum (labels above the spectrum) are shifted by 328 ns, the time separation between the two ALS bunches. The maximum time resolution for the experiment can be determined from the width of the prompt peaks resulting from light scattered from the sample onto the detector. These peaks, which actually have a flight time near zero, have a full width at half maximum (FWHM) of only 200 ps. The most intense peak in the spectrum,  $\text{Ar}^{+4}$ , shows a FWHM of 840 ps, while  $\text{Ar}^{+7}$ , which is the most efficiently space-focused peak, shows a FWHM of 600 ps. The high degree of time resolution observed is a result of the excellent timing characteristics of the ALS, proper handling of the timing signal in the ion-TOF analyzer and associated electronics, and the optimization of the analyzer space-focusing parameters.

#### 4. Summary

We have reported on the commissioning of beamline 9.3.1 at the ALS. This beamline is a windowless beamline at ALS, covering the 1-6 keV photon-energy range. The beamline is designed to achieve the goals of high flux, and preservation of the high brightness from ALS. To achieve these goals, two novel technical designs were used; an unusual optical design for x-rays beamlines, in which matched toroidal mirrors are positioned before and after the double-crystal monochromator, and a new "Cowan type" double-crystal monochromator was implemented. The beamline was commissioned in the spring of 1995, with a prototype M1 mirror with a fixed radius but with the M2 mirror not installed. Not having M2 mirror substantially lowered the flux delivered to a small sample area. We discussed the initial operation of the beamline in this configuration. We presented absorption spectrum of Cl, S, and Ar for monochromator calibration and first TOF data from the beamline. The initial data demonstrated the resolution and stability of the beamline and it provides an indication of the expected performance when the M2 mirror is installed.

## Acknowledgments

This work was supported by the Director, Office of Energy Research, Office of Basic Energy Sciences, Materials Sciences Division of the U.S. Department of Energy under Contract No. DE-AC03-76SF00098. This work is also partially supported by an award from Research Corporation. Acknowledgment is made to the donors of the Petroleum Research Fund, administered by the ACS. In addition, we received partial support from the university research grants and fellowships committee of the University of Nevada, Las Vegas. TOF measurements were performed using a spectrometer based upon work support by the National Science Foundation under Grant No PHY-9303916. We would like to thank P.L. Cowan for his help and encouragement in the development of this monochromator, Steward Ryce for actuarially building the monochromator and E.M. Chow for assistance in ray tracing the optical system. We would also like to thank the staff at the ALS and D. Chen, G. Fisher, M. Thomas, J. Kring, and K. Batson for their assistance in different parts of the commissioning of the beamline.

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### Captions

Table 1 Range and resolution of M1 and M2 mirror chambers in the X, Y, Z direction, as well as rotation about these axis.

Fig. 1. The Ar K absorption spectrum was acquired with 5 torr of Ar. The data was not normalized, and the feedback system was not used.

Fig. 2 Absorption spectrum of the Cl K edge of 4 torr of  $\text{CF}_3\text{Cl}$ .

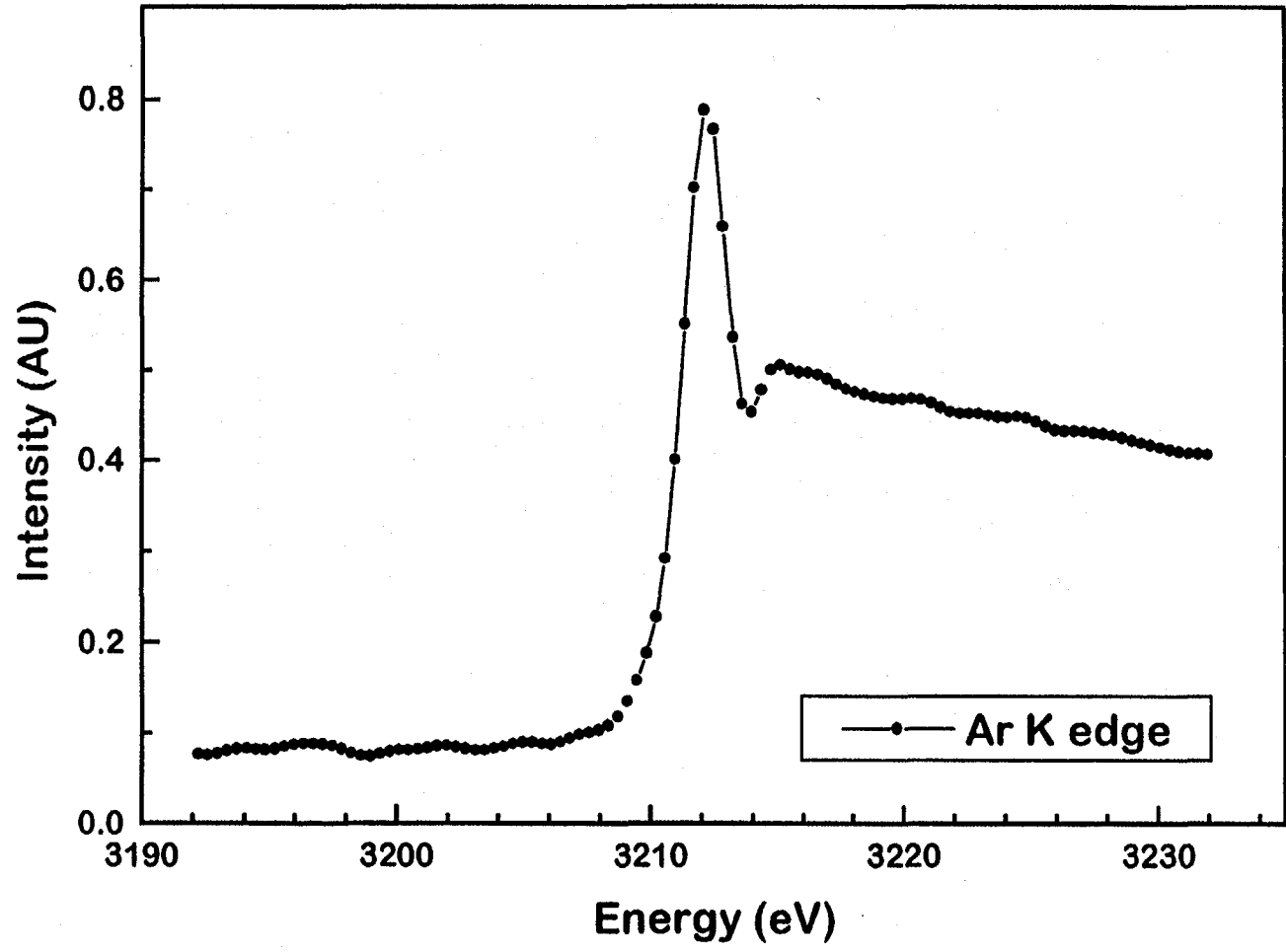
Fig. 3 Sulfur K absorption spectra acquired with 5 torr of  $\text{SF}_6$ . The scale for the upper spectrum has been increased 8.5 times to show the smaller structures that were not visible in the lower spectrum.

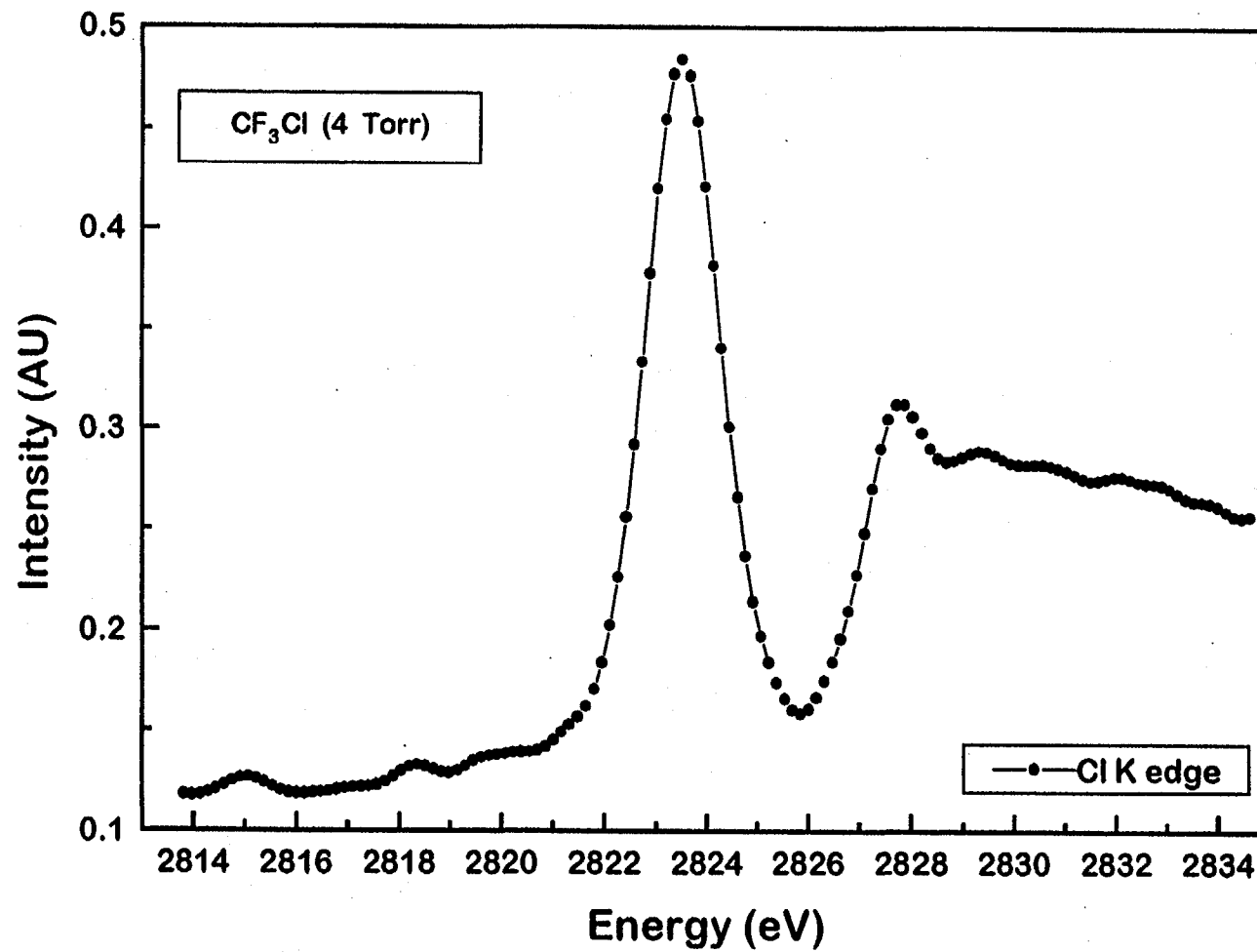
Fig. 4 This figure shows a time of flight spectrum taken at a photon energy above the argon K-shell threshold (3205 eV). The data were collected using a space-focused ion time-of-flight mass spectrometer. Because of the double-bunch mode of operation, two spectra are shown in Fig. 4, indicated by labels above or below the baseline, respectively.

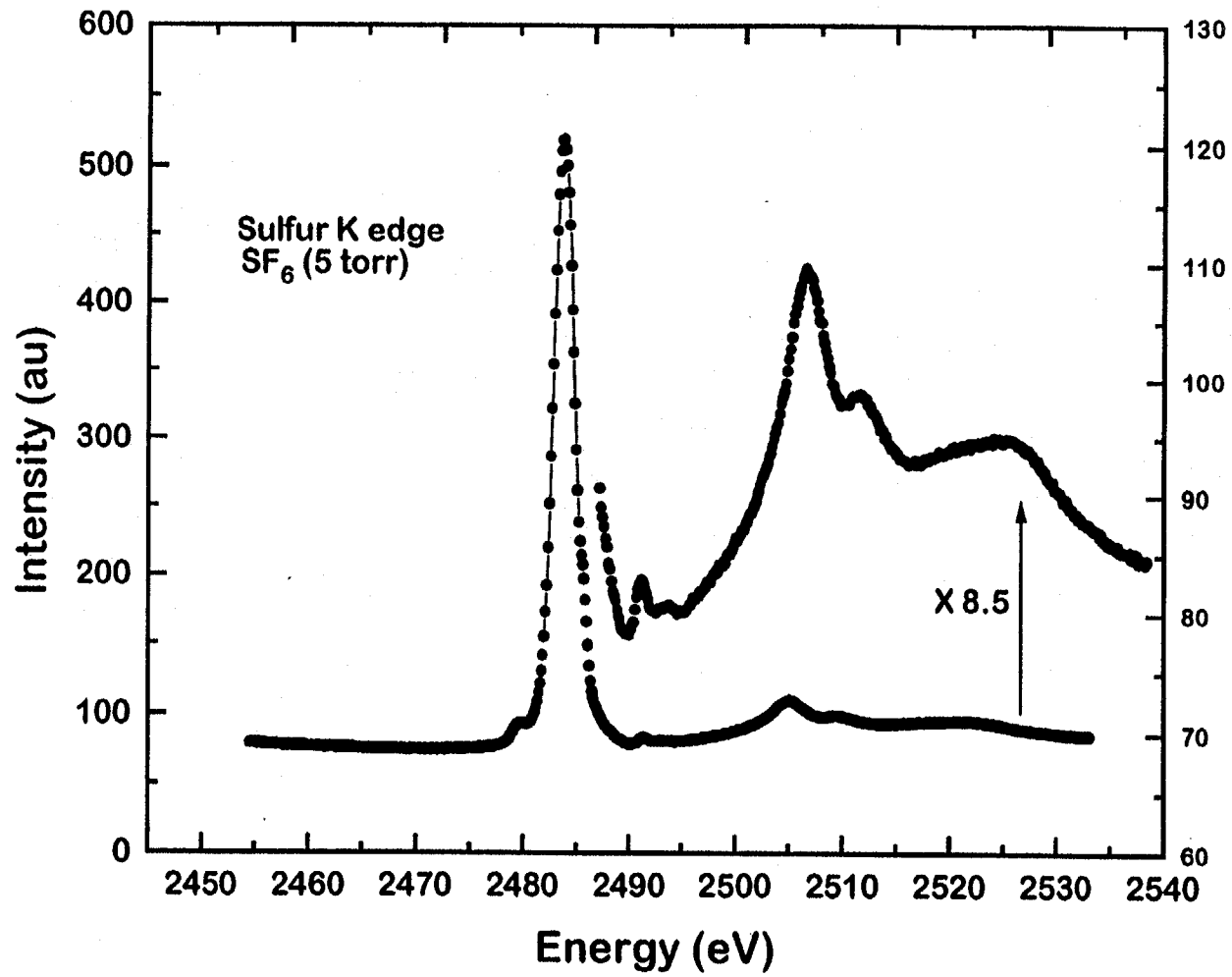
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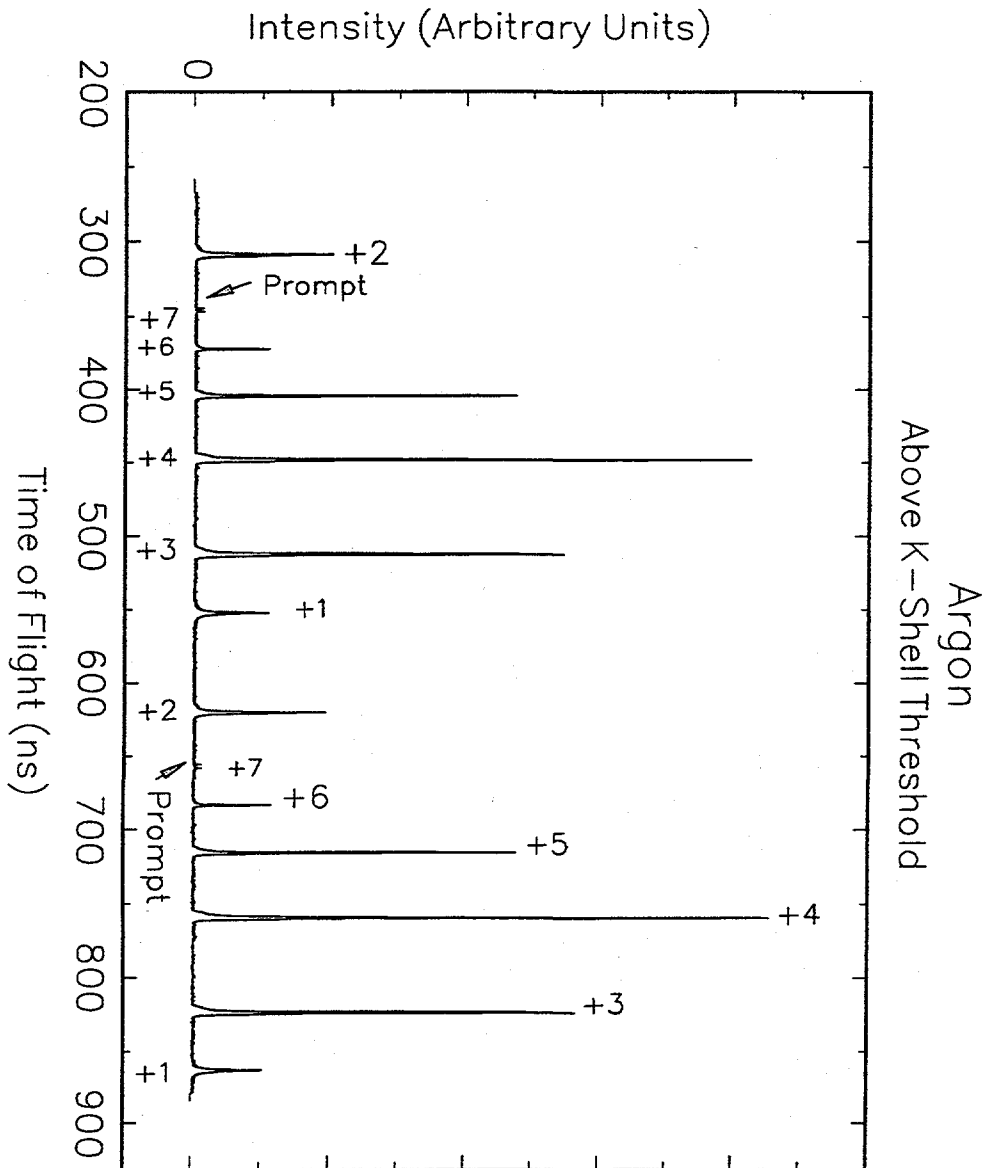
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X	10mm	10 $\mu\text{m}$
Y	20mm	0.1mm
Z	20mm	5 $\mu\text{m}$
rX	8°	1 $\mu\text{rad}$
rY	1°	0.01°
rZ	1°	0.005°

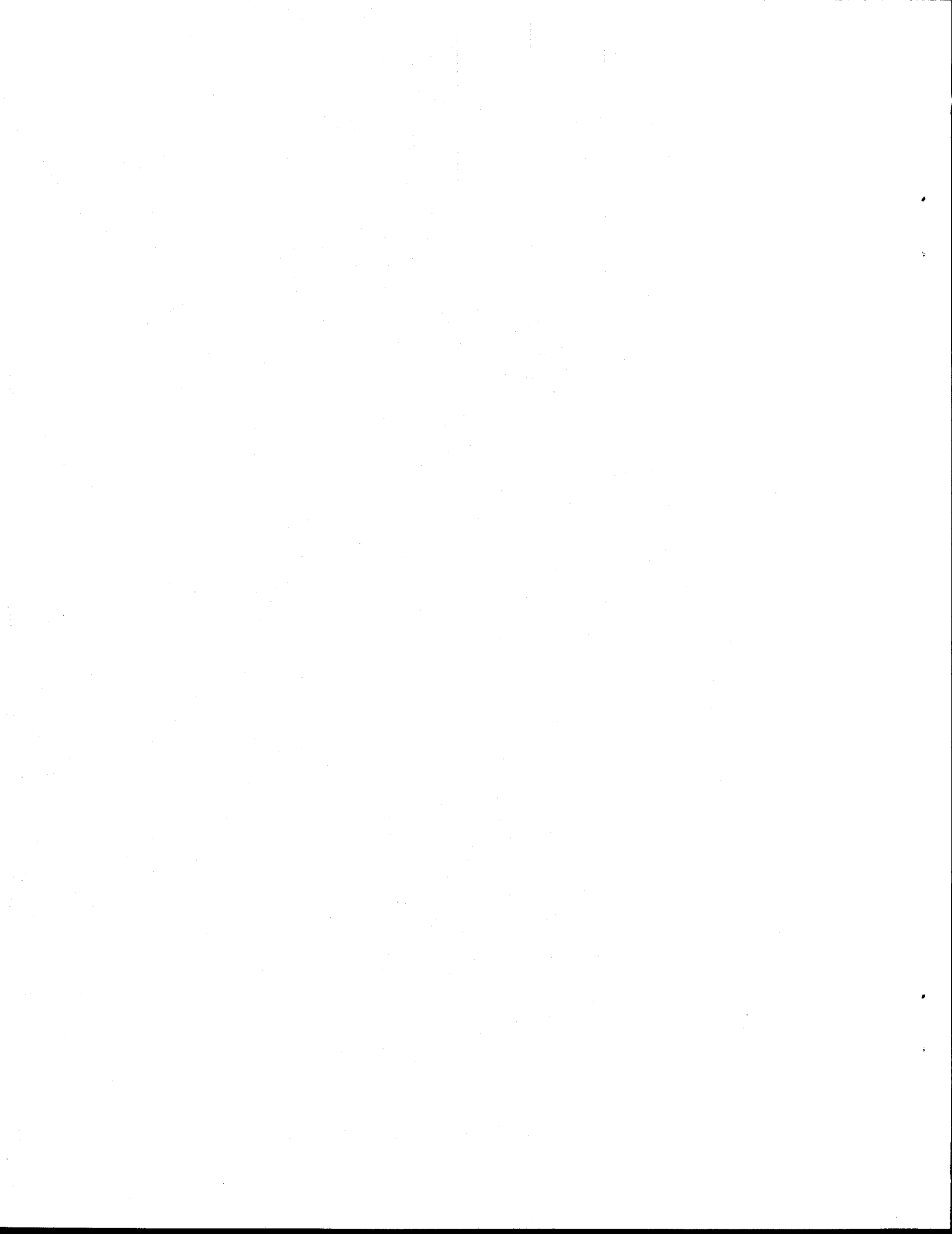
Table 1



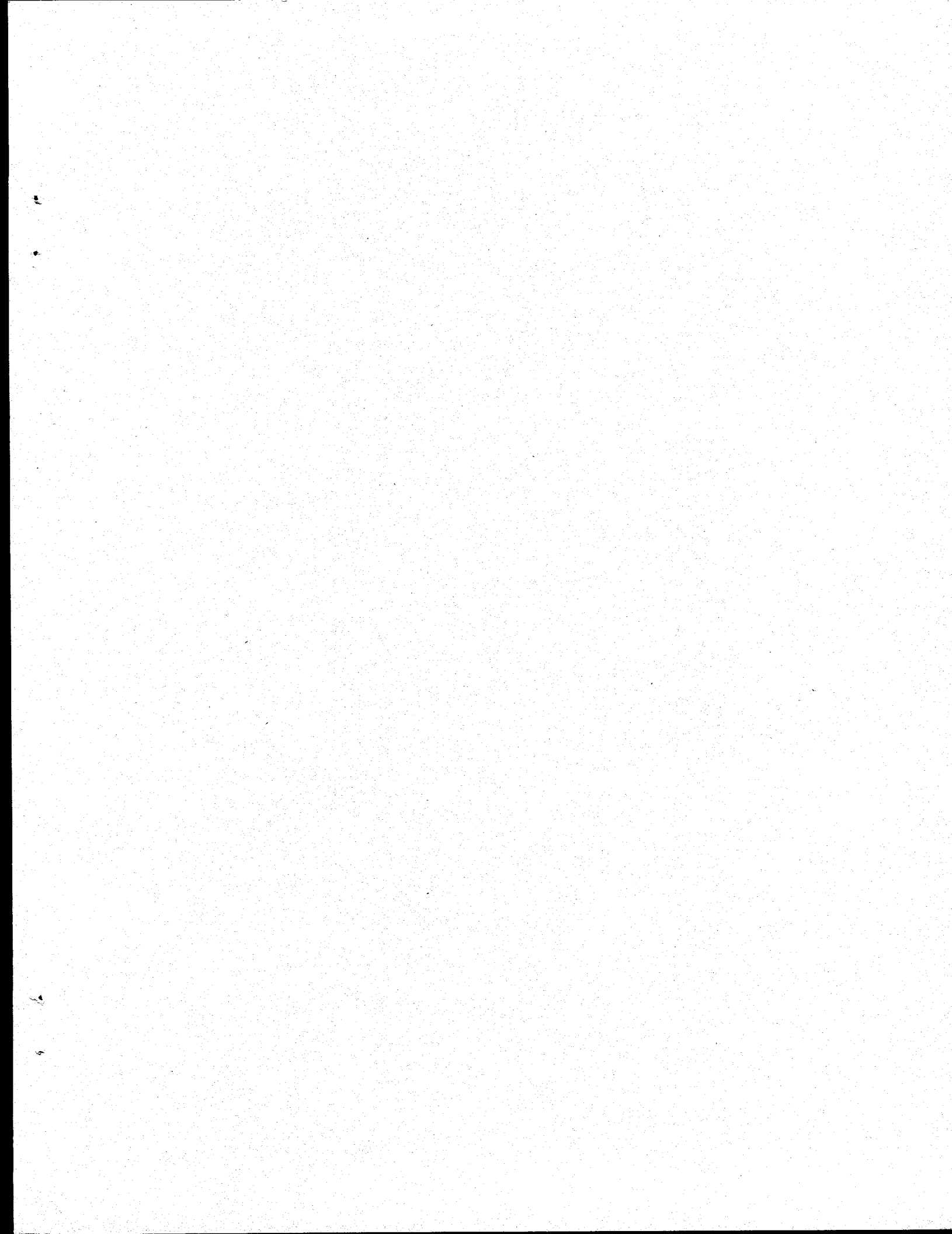












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