NEUTRON SCATTERING AT THE HIGH FLUX ISOTOPE REACTOR AT OAK RIDGE NATIONAL LABORATORY

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ABSTRACT

Since its beginnings in Oak Ridge and Argonne in the late 1940's, neutron scattering has been established as the premier tool to study matter in its various states. Since the thermal neutron wavelength is of the same order of magnitude as typical atomic spacings and because they have comparable energies to those of atomic excitations in solids, both structure and dynamics of matter can be studied via neutron scattering. The High Flux Isotope Reactor (HFIR) provides an intense source of neutrons with which to carry out these measurements. This paper summarizes the available neutron scattering facilities at the HFIR.

Introduction

The de Broglie wavelength of thermal neutrons is comparable to atomic spacings. In addition, neutron and phonons have energies of the same order of magnitude. These two properties alone make neutrons an ideal probe of the structure and dynamics of condensed matter systems. But neutrons have other characteristics that make them even more useful. Its magnetic moment makes it possible to study magnetic structure and dynamics, along with its nuclear counterpart. They interact strongly with nuclei, the strength of which varies dramatically and non-monotonically from nucleus to nucleus. It is possible to study light elements and isotopic variations of scattering lengths can be used to advantage; for example, the contrast between hydrogen and deuterium has revolutionized polymer physics. Neutrons are highly penetrating (with few exceptions) since they have no charge, thus they can be used to study bulk materials nondestructively. This is of obvious interest in industrial applications.

The High Flux Isotope Reactor (HFIR) at Oak Ridge is the highest flux research reactor in the world. Running at a power level of 85MW, the flux outside the beryllium reflectors (which surround the core) is 1.5×10^{15} neutrons/cm²/sec. While the HFIR was built primarily to produce transplutonic isotopes, its high neutron flux in the thermal range makes it especially suited for neutron scattering research as well as neutron activation analysis and materials irradiation. It is noteworthy that the HFIR is the only source in the western hemisphere of Cf-252 and high-specific activity Co-60 isotopes, which are used widely in medical and research applications.

Twelve instruments, located on the four HFIR beam lines, are dedicated to neutron scattering research in various fields of physics, chemistry, biology and materials science. The beam room layout is shown in Fig. 1. The instruments at the HFIR are varied in their applications and are described in detail in the remainder of this article. They include four triple-axis spectrometers (TAS), a four circle diffractometer, a reflectometer, a medium-resolution double crystal small-angle neutron scattering (SANS) spectrometer, a wide angle neutron diffractometer (WAND), a powder diffraction instrument and a 30-m small-angle

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Figure 1. The layout of the neutron scattering instruments in the HFIR beam room.

scattering facility. The HFIR is a national user facility and its neutron scattering instruments are available to qualified users on a competitive proposal¹ basis.

1. Triple Axis Spectrometers

There are four triple axis spectrometers (TAS) at the HFIR, one of which can be used in the polarized beam mode with full polarization analysis. The monochromator systems for these instruments are different for each individual spectrometer and are listed below. The analyzers, on the other hand, can be selected from an array common to the four spectrometers which includes germanium, silicon and beryllium crystals and both flat and horizontally-focusing pyrolytic graphite (PG). The energy resolution ΔE for these instruments depends on the individual setup. Typical values of $\Delta E/E_i$ at the elastic position are:

2-10% for $E_i = 14.7$ meV and PG(002) monochromator

1-3% for $E_i = 14.7$ meV and Be(1 0 1) monochromator, and

1.5-6% for $E_i = 30$ meV and Be (1 0 1) monochromator.

It is difficult to estimate the typical time required to do a three-axis experiment since the complexity of the measurement varies dramatically. For instance, the number of phonon modes varies as $3n$, where n is the number of atoms per unit cell. The total scattering power can thus be concentrated in as few as three branches, which will make them easier to measure. An example of this is elemental copper, where all the modes can be mapped out throughout the Brillouin zone overnight. The required resolution and the sample size also factor into determining counting times. In general, single crystals should be as large as possible; since phonon cross-sections are orders of magnitude smaller than Bragg scattering, extinction is not a problem. Recent magnetic measurements carried out on the TAS instruments at HFIR include magnons² in "itinerant" triangular antiferromagnets Mn_3Sn and Mn_3Ge , a polarized neutron study of magnetic excitations³ in YBCO, excitations in quasi-one-dimensional, dilute antiferromagnet $CsCo_xMg_{1-x}Br_3$ ⁴ and in the 3-D Ising magnet $Fe_xZn_{1-x}F_2$ ⁵, and fracton excitations in the Heisenberg antiferromagnet⁶ $RbMn_xMg_{1-x}F_3$ near the percolation concentration. Phonons in BSCCO⁷, lattice instability in β -AgZn⁸, and lattice dynamics in Cu-Al-Be alloys⁹ are among the recent inelastic nuclear experiments.

Analyzer crystals for the Triple-Axis Spectrometers

Be (*h* 0 *l*)

PG (0 0 2), both flat and horizontally focusing

Si(1 1 1)

Ge(1 1 1)

⁵⁷Fe(Si) (1 1 0) and Heusler (1 1 1) analyzers are available for polarized beam analysis on HB1.

Three of these spectrometers are controlled by VAX stations. The remaining one is controlled by a PDP computer which will be soon replaced by a VAX station. Utilities are available to calculate resolution functions and make intensity comparisons between different geometries.

The second and third axes of all the TAS (except HB1A) are currently being rebuilt to enhance the performance and reliability of these machines. The new version incorporates conical gears which will allow for very accurate positioning of the motors while maintaining the original cantilever design. The backlash is much less than 0.005° , which is substantially better than other designs.

A variety of ancillary equipment for special sample environment is available for TAS. Most of them can also be accommodated on the other instruments. Cryomagnets with both vertical and horizontal field up to several Tesla, cryostats, closed cycle refrigerators and a dilution refrigerator can maintain sample environments from 100 mK to room temperature via computer control. Furnaces extend the temperature range available to 1900K. Also, a clamp type pressure cell (to 3 GPa) is available.

HB1 This instrument can be operated either as a polarized or as an unpolarized triple-axis spectrometer. Magnetic scattering involves the flipping of the neutron's spin whereas nuclear scattering does not. The polarization state of the neutron beam is controlled by a spin-flipping device (Mezei coil) that can be placed either in the incident and/or scattered beam. The spin flippers are computer controlled and the polarization states can be changed at any time during the experiment. The selection of any of three monochromator crystals can be performed remotely via computer control (see table below).

HB1 Triple-Axis Spectrometer parameters

Monochromators	$^{57}\text{Fe}(\text{Si})$ (1 1 0) Be (1 0 1) PG (0 0 2)	Polarized beam
Monochromator angles	$10^\circ < 2\Theta_M < 95^\circ$	
Analyzer angles	$-120^\circ < 2\Theta_A < 120^\circ$	
Scattering angles	$-60^\circ < \Phi < 120^\circ$ $-60^\circ < \Phi < 60^\circ$	With an electromagnet
Sample angle	$0^\circ < \Psi < 360^\circ$	
Collimations (FWHM)	C1: open beam tube=60' C2: 10', 20', 40' C3: 10', 20', 40' C4: 20', 40', 60', 120'	in pile Monochromator-sample Sample-analyzer Analyzer-detector
Beam size	$3.75 \times 6 \text{ cm}^2$ maximum	
Filters	Sapphire PG	In pile
Flux at sample	3.5×10^6 neutron/cm ² /s $^{57}\text{Fe}(\text{Si})$ polarizing monochromator and C2=40'	at 1.28 Å

HB1A This instrument was constructed through a collaboration with Ames Laboratory at Iowa State University. HB1A has a fixed incident energy of 14.7 meV. The monochromator is a double crystal system with a vertically focusing PG monochromator in the main beam of HB-1. At the second monochromator position, two PG monochromators are available: a vertically focusing unit and a double focusing unit. Sample size and experimental requirements dictate which monochromator is used. The double bounce configuration in close proximity to the beam tube (no monochromator drum) provides one

of the most intense beams at 14.7 meV at the HFIR and the cleanest beam in terms of higher order contamination. Any of the array of analyzers available for all triple axis spectrometers can be used on this spectrometer.

HB2 This is a very flexible triple axis spectrometer which allows the choice of three vertically focusing monochromator crystals (Be, Si and pyrolitic graphite) as well as a flat Be monochromator. This instrument also provides in pile cold beryllium or silicon filters and premonochromator collimation.

HB1A Triple-Axis Spectrometer parameters

Monochromator	PG (0 0 2)	double crystal
Monochromator angle	$2\Theta_M$ fixed at 41.2°	$E_i=14.7$ meV
Analyzer angles	$-10^\circ < 2\Theta_A < 120^\circ$	
Scattering angles	$0^\circ < \Phi < 150^\circ$	
Sample angle	$0^\circ < \Psi < 360^\circ$	
Collimations (FWHM)	C1: open beam tube=60' C2: 10', 20', 30', open = 40' C3: 10', 20', 30', 40' C4: 35', 70', 105'	in pile Monochromator-sample Sample-analyzer Analyzer-detector
Beam size	3.75×5 cm ² maximum	
Filters	Sapphire HOPG ($\lambda/2 \approx 10^{-4} \lambda$)	In pile between monochromators
Flux at sample	2×10^7 neutron/cm ² /s	at 2.35Å with C2=40'

HB2 Triple-Axis Spectrometer parameters

Monochromators	Be (1 0 1) PG (0 0 2) Si(1 1 1) Be (1 0 3)	Vertically focusing Vertically focusing Vertically focusing Flat
Monochromator angles	$10^\circ < 2\Theta_M < 100^\circ$	
Analyzer angles	$-120^\circ < 2\Theta_A < 120^\circ$	
Scattering angles	$-50^\circ < \Phi < 130^\circ$	
Sample angles	$0^\circ < \Psi < 360^\circ$	
Collimations (FWHM)	C1: 20', 40', open =60' C2: 10' to 60' in 10' steps C3: 10', 20', 40', 80' C4: 40', 60', 120'	in pile Monochromator-sample Sample-analyzer Analyzer-detector
Beam size	3.75×5 cm ² maximum	
Filters	Silicon (cold) Beryllium (cold) PG	in pile in pile
Flux at sample	3×10^7 neutron/cm ² /s PG monochromator, PG filter ,	at 1.4Å C1=60', C2=40'

HB2 This is a very flexible triple axis spectrometer which allows the choice of three vertically focusing monochromator crystals (Be, Si and pyrolytic graphite) as well as a flat Be monochromator. This instrument also provides in pile cold beryllium or silicon filters and premonochromator collimation.

HB3 This instrument provides two choices of monochromators, namely Be(*h* 0 *l*) and pyrolytic graphite (0 0 2). This spectrometer also provides an in pile sapphire filter and choice of pre-monochromator collimators.

HB3 Triple-Axis Spectrometer parameters

Monochromators	Be (<i>h</i> 0 <i>l</i>)	
	PG (0 0 2)	
Monochromator angles	$10^\circ < 2\Theta_M < 90^\circ$	
Analyzer angles	$-120^\circ < 2\Theta_A < 120^\circ$	
Scattering angles	$-60^\circ < \Phi < 120^\circ$	
Sample angle	$0^\circ < \Psi < 360^\circ$	
Collimations (FWHM)	C1: 20', 40', open =60'	in pile
	C2: 10' to 60' in 10' steps	Monochromator-sample
	C3: 10', 20', 40', 80'	Sample-analyzer
	C4: 20', 40', 60', 120'	Analyzer-detector
Beam size	$3.75 \times 5 \text{ cm}^2$ maximum	
Filters	Sapphire	In pile (Optional)
	PG	
Flux at sample	1×10^7 neutron/cm ² /s	at 1.4 Å
	Be (0 0 2) monochromator, PG filter	C1=C2=40',

2. Diffractometers

There are three neutron diffractometers at the HFIR, a four circle diffractometer for the study of single crystals, a high resolution powder diffractometer, and a Wide Angle Neutron Diffractometer (WAND) that can be used for the study of single crystals as well as polycrystalline specimens.

Residual Stress (HB-2 in Two-Axis Operation)

The HB2 triple-axis spectrometer can be used as a residual stress diffractometer. A linear position sensitive detector is mounted in the location of the energy analyzing crystal. The addition of incident and receiving collimators and sample positioning translators completes the conversion of the TAS to the stress machine. Typical accuracy in strain determinations is 0.5 to 1.0×10^{-4} . Samples weighing up to 10 kilograms and with dimensions up to 50 cms are typical. Samples up to 150 kilograms supported by a lifting bar can also be accommodated. A small tensile frame with a 4000 Newton capacity is available for elastic constant measurements.

Four Circle Diffractometer (HB2A) This single crystal diffractometer has been recently upgraded. A Huber 4-circle diffractometer has been interfaced to a Crystal Logic controller and a DEC Alpha workstation which supports the UCLA system of crystallographic software. A 2-inch diameter ^3He end-on cylindrical detector contains seven equal-area chambers, arranged hexagonally, with each connected to a separate counting channel. In a typical application the center chamber is used to measure a Bragg peak while the background is measured in the six surrounding chambers. A closed cycle refrigerator is used to cool the specimens under study down to 15 K.

HB2A Four Circle Diffractometer parameters

Monochromator	Ge (<i>h h l</i>)	Flat
Monochromator angle	$2\Theta_M$ fixed at 45.5°	$\lambda = 0.84, 0.89, 1.00,$ $1.32, 1.55$ or $2.5 \text{ \AA}$
Scattering angles	$-100^\circ < 2\Theta < 100^\circ$ $-50^\circ < \omega < 50^\circ$ $0^\circ < \chi < 360^\circ$ $0^\circ < \phi < 360^\circ$	
Beam size	0.6 cm diameter	
Maximum crystal size	0.5 cm diameter	
Maximum unit cell size	30 $\text{\AA}$	
Flux at sample	8×10^6 neutron/cm ² /s for a wavelength of 2.5 $\text{\AA}$	

High Resolution Powder Diffractometer (HB4) The high-resolution neutron powder diffractometer has been operating since 1991. Upgrades to hardware and software are ongoing; however, this instrument has already demonstrated itself as an important addition to the expanding repertoire of experimental tools available at the HFIR.

Presently, the monochromator is a (115) Ge crystal, which can be oriented to select incident wavelengths of 1.0, 1.4, 2.2, and 4.2 $\text{\AA}$. An array of 32 equally spaced (2.7°) ^3He detectors, each with a 6' mylar foil Soller collimator, can be step-scanned over a range of up to 40° for scattering angles between 11° and 135° . Soller slit collimators of 12' and 20' are positioned before and after the monochromator crystal, respectively. The design parameters give a resolution minimum of $\Delta d/d = 2 \times 10^{-3}$ at about $70^\circ 2\Theta$. A 15-element focusing (117) Ge monochromator, which will provide greater intensity, is under construction.

Future renovations will include two new in-pile collimators, selectable by the operator to provide different resolutions. The height of the primary beam incident on the monochromator will be enlarged. The take-off angle from the monochromator will be increased to 130° , to improve the higher angle resolution. The orientation of the new focusing monochromator at this take-off angle will provide a 1.4 $\text{\AA}$ wavelength beam. Wavelengths of 1.1, 1.9, and 3.0 $\text{\AA}$ will also be available from different (*hhl*) planes, by small rotations ($< 10^\circ$) of the monochromator.

The diffractometer control software is user-friendly and operates presently on a VAX computer (running VMS), which is part of a network. A complete diffraction pattern can

be obtained in a few hours depending upon the chosen 2θ range, step size and counting time. The status of the diffractometer can be checked remotely.

A variety of special environment devices are available to provide sample temperatures between 1.8 K to 1900 K. A clamp-type high pressure cell can provide pressures up to 3 GPa. A selection of vanadium and aluminum cans are available, as well as the ability to spin or oscillate the sample to reduce preferred orientation effects. The volume of a sample needed to do a measurement depends on the scattering power of the sample, but presently is in the range of 0.25 to 3 cm³. The high-pressure cell accommodates a 0.25 cm³ size sample.

Local utility programs provide for instrumental calibration corrections, statistical check for noisy data points, individual detector displays, peak-fitting, pattern addition, pattern archiving and retrieval. A full range of software for Rietveld structure analysis, unit cell refinement, and various crystallographic computations is also available.

Recent experiments performed on this instrument have included studies of the structures of the cuprate superconductors both under high pressure¹⁰ and ambient pressure^{11,12}, phase transformations and residual microstresses in ceramic composites¹³ crystal structure - property relationships in oxides¹⁴, nuclear and magnetic structures of other interesting oxides^{15,16,17,18}.

HB4 High-Resolution Powder Diffractometer parameters

Monochromator*	Ge (1 1 5)	Flat
Monochromator angles*		$2\theta_M = 80^\circ$ $\lambda = 1.4 \text{ \AA}$
Scattering angles	$11^\circ < \Phi < 135^\circ$	
Sample angle(S)	$0^\circ < \Psi < 360^\circ$	
Collimations (FWHM)	C1: 12' C2: 20' C3: 6'	in pile Monochromator-sample Sample-detector
Beam size*	$3.75 \times 5 \text{ cm}^2$ maximum	
Flux at sample*	2×10^5 neutron/cm ² /s	

* To be modified during renovation.

HB4A Wide Angle Neutron Diffractometer parameters

Monochromator	Be(1 0 1)	Flat
Monochromator angle	$2\theta_M = 52.5^\circ$	$\lambda = 1.54 \text{ \AA}$
Scattering angles	$10^\circ < \Phi < 140^\circ$	
Sample angles	$0^\circ < \Psi < 360^\circ$	
Beam size	$3.75 \times 5 \text{ cm}^2$ maximum	
Flux at sample	2×10^6 neutron/cm ² /s	

Wide Angle Neutron Diffractometer WAND (HB4A) This instrument was designed to provide two specialized data collection capabilities, (1) fast time-resolved measurements of powder diffraction patterns, and (2) measurements of diffuse scattering in single crystals using the flat-cone geometry. These capabilities are achieved with the use of a linear

position sensitive detector that subtends a 130° scattering angle with an angular resolution of about 0.6° . This detector can be tilted off the horizontal plane (around an axis perpendicular to the incident beam) for single crystal studies. The untilted detector configuration can be used for powder diffraction experiments. A Be(1 0 1) monochromator provides neutrons with a wavelength of $1.54 \text{ \AA}$. An oscillating collimator system, placed between the sample and detector, reduces the background due to the sample environment. The WAND is operated in collaboration with the Japan Atomic Research Institute (JAERI) under the US-Japan Cooperative Program on Neutron Scattering Research.

3. Small -Q Spectrometers

Four instruments at the HFIR are designed to study neutron scattering at small wavevectors Q . Of these a 30 m Small Angle Neutron Scattering (SANS) instrument has been available to the user community for some time. A neutron reflectometer has been recently tested and will soon be available to users. Two double crystal instruments, which will provide medium and high- Q resolution share the HB3A beam line with the reflectometer. The medium resolution instrument has just been completed and is in the testing phase, the ultra-high resolution double crystal SANS spectrometer is in the design stage. When complete, the three SANS machines will be able to probe structures on a length scale with a wide range from $20 \text{ \AA}$ to greater than $20000 \text{ \AA}$. Ancillary equipment on the SANS include furnaces, cryomagnets, cryostats, closed cycle and dilution refrigerators, goniometers, sample changers and Couette and Poiseuille shear cells.

30 m SANS This instrument, which was built with funds supplied by the National Science Foundation, utilizes a pinhole geometry with collimating slits of $0.5 - 3.5 \text{ cm}$ dia. separated by a distance of 10 m ¹⁹. The beam may be transported to distances of $1.5, 3.5$ or 7.5 m from the sample by means of moveable neutron guides. The detector, with an active area of $64 \text{ by } 64 \text{ cm}^2$ and resolution element dimensions of $1.0 \text{ by } 1.0 \text{ cm}^2$, can be positioned at any distance from 1.5 to 19 m from the specimen by moving a motor-driven detector carrier along rails in an evacuated flight path. The standard incident wavelength provided by a bank of pyrolytic graphite crystals, is $4.75 \text{ \AA}$, though this can be changed to $2.38 \text{ \AA}$ by substituting graphite for the cold beryllium filter normally in position. This gives a Q -range of $0.003 < Q < 0.75 \text{ \AA}^{-1}$ and the instrument is capable of resolving scattering from particles containing as few as 60 atoms.²⁰ The specimen chamber can be fitted with any of the ancillary equipment. The temperature and sample position of a four-position furnace may be controlled via IEEE-488 computer interface from the system's computer. This equipment is used principally for polymer research in the range $40 < T < 350^\circ\text{C}$. A programmable 6-position sample changer suitable for liquid systems (e.g. micelles, microemulsions, biological macromolecules in solution etc.) is also available with temperature control in the range $-5 < T < 85^\circ\text{C}$. All data may be converted to an absolute ($\pm 4\%$) differential cross section per unit sample volume (in units of cm^{-1}) by comparison with pre-calibrated secondary standards (provided by the Center), based on the measurement of beam flux, vanadium incoherent cross section, the scattering from water and other reference materials²¹. The detector efficiency calibration is based on the scattering from light water and this leads to angle-independent scattering for vanadium, H-polymer blanks and water samples of different thicknesses in the range $1\text{-}10 \text{ mm}$. Users may take away data in the form of absolutely calibrated 1-D ASCII files along with a standard plotting program. This instrument is part of the Center of Small Angle Scattering Research, which also includes a 10-m small-angle X-ray scattering spectrometer²².

SANS parameters

Q-range	$0.003 < Q < 0.75 \text{ \AA}^{-1}$
Q resolution:	10^{-3} to $10^{-1} \text{ \AA}^{-1}$, depending on instrumental settings
Beam size	0.25 - 3.0 cm (diameter)
Flux	up to 5×10^5 neutron/cm ² /s (with all 3 beam guides in place)

Neutron Reflectometer²³ This instrument has recently been relocated to the fixed wavelength beam line HB3A, where a pyrolytic graphite monochromator provides an intense incident beam. The instrument uses a position-sensitive detector to track specular and off-specular scattering in a horizontal plane. Reflectivities down to about 2×10^{-6} may be measured with data collection times of a few hours. The standard sample position holder accommodates flats of diameters up to 10 cm. Static and Poiseuille shear liquid-solid (quartz and sapphire) interface cells may also be mounted on this stage.

Reflectometer parameters

Wavelength	2.59 Å	fixed
Flux at sample	5×10^5 neutron/cm ² /s	
Beam size	0.2 × 2 cm ²	
Q range	$\sim 0.005 < Q < 0.16 \text{ \AA}^{-1}$	
Minimum Reflectivity	$\sim 2 \times 10^{-6}$	

Medium-resolution double crystal spectrometer parameters

Wavelength	2.59 Å	fixed
Q range	$8 \times 10^{-4} < Q < 3 \times 10^{-2} \text{ \AA}^{-1}$	
Q resolution:	6×10^{-4} or $3 \times 10^{-3} \text{ \AA}^{-1}$	not continuously variable
Beam size	1 cm wide × 5 cm high	at sample position, maximum

Medium-Resolution Double Crystal spectrometer This instrument uses two spherically bent silicon crystals as monochromator and analyzer. The bent crystals coarsen the resolution while dramatically improving the peak intensity. The curvature of these crystals has the added benefit of focusing the beam spatially, which allows the use of small samples. In its current geometry, the Q-resolution is $0.0006 \text{ \AA}^{-1}$. A Q of $0.001 \text{ \AA}^{-1}$ corresponds to structures greater than 6000 Å in size. The resolution and degree of spatial focusing can be varied, although not continuously, with the choice of analyzer reflection, curvature of the crystals and monochromator-analyzer distance. The peak intensity without any sample (and with the largest aperture) is greater than 10^5 counts/s at 2.59 Å. The sample can be rotated to study angular dependences in the scattering. This

spectrometer can accommodate any of the available special environment apparatus (including Displex refrigerators, cryomagnets, furnaces, et cetera).

High-resolution double crystal spectrometer parameters

Wavelength	2.59 Å	fixed
Q-range	$4 \times 10^{-5} < Q < 3 \times 10^{-3} \text{ Å}^{-1}$	
Beam size	$2 \times 4 \text{ cm}^2$ maximum	

Ultrahigh-Resolution Double crystal spectrometer This small-angle scattering spectrometer will use two multiple bounce channel cut silicon crystals. The multiple bounces in the monochromator and analyzer will reduce the scattering in the wings of the main peak while maintaining the same peak intensity. This will allow measurements to be made at a lower Q without interference from the central peak. This instrument will provide a very high angular resolution in the scattering plane and will provide access to wavevectors Q as small as $4 \times 10^{-5} \text{ Å}^{-1}$. This machine is presently under design and construction and will become available to the user community in the latter half of 1996. 5

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