ENRAF NONIUS DELFT

CAD 4 Kappa Axis Diffractometers

CAD 4

CAD 4 F

CAD 4 SDP

More than 50 years experience in x-ray diffraction

Introduction

Since its introduction, the CAD 4 Kappa Diffractometer has become the outstanding system available. The diffractometer design opened new possibilities of accuracy, reliability and simplicity of operation to the crystallographer, both in its hardware and software. The use of Kappa Geometry not only provides a completely open system, eliminating the hardware obscuration of a chi circle, but provides, in addition, the highest possible accuracy which is maintained over the long life of the instrument.

There is ample space to work with all necessary accessories like high and low temperature devices, low or high pressure cells and others. In most cases, obscurations from these devices are either avoided or greatly reduced using the Kappa Geometry. The system permits the crystallographer a choice of whether to work with an almost completely automatic system or to utilize its many facilities for intensive crystal study and investigation.

The revolutionary goniometer is coupled with an outstandingly high-stability X-ray generator and computer control equipment ranging from the most basic to the most sophisticated system.

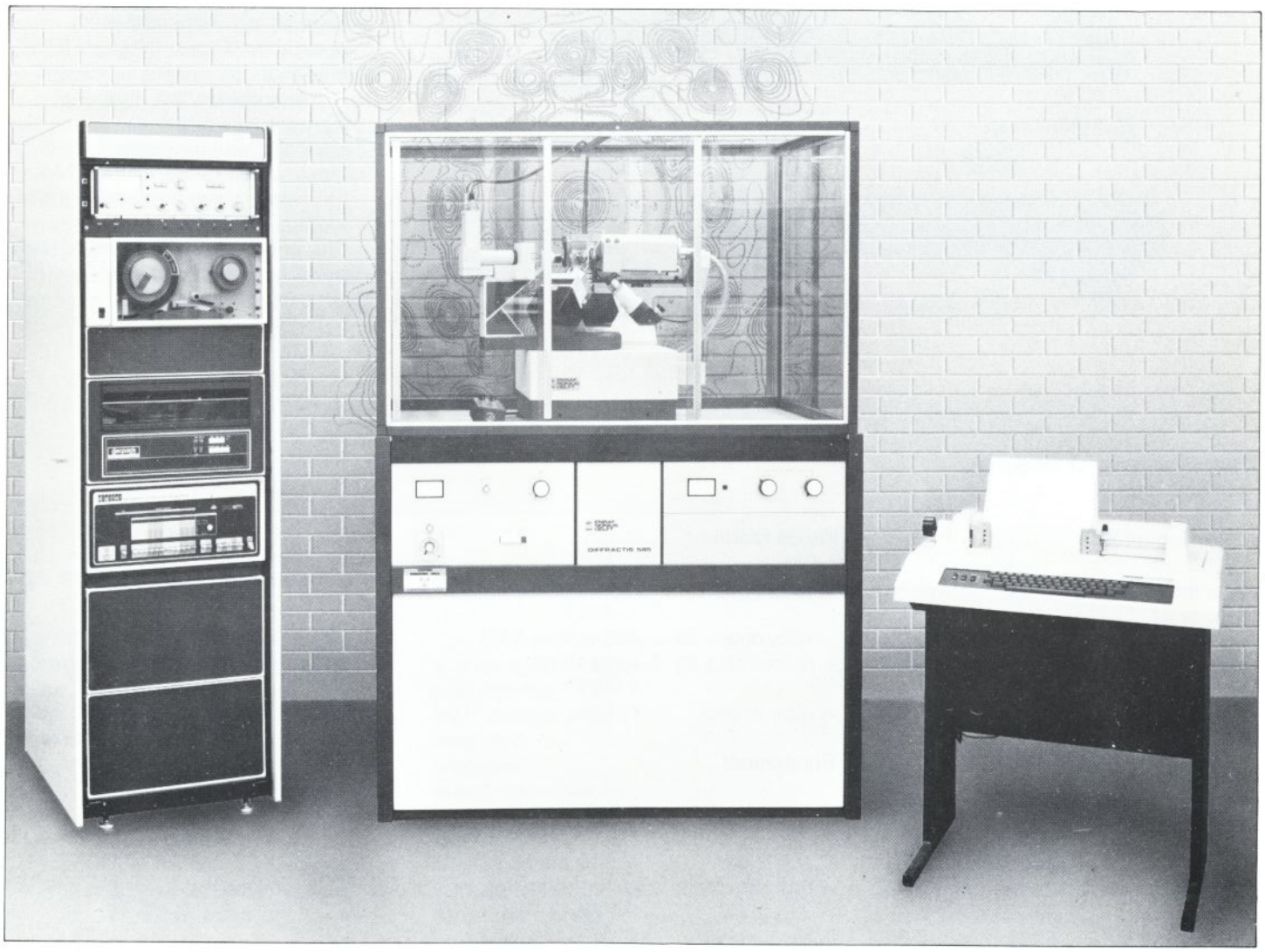
Complete software systems have been developed which are provided at no additional cost. All programs offer the user a wide choice in planning experiments.

The basic CAD 4 system includes a PDP-8 computer with 8K of core memory, 1.6 million words disk, read write magnetic tape and DECwriter terminal.
With a slightly expanded PDP-8 computer control programs are available in FORTRAN IV language. For only a minor difference in price this CAD 4 F offers the most complete software system ever developed for a commercial single crystal diffractometer and at the same time it supplies a possibility for the crystallographers to add their own ideas and programs.
Optional systems include control by powerful computing systems, like the PDP-11/34 and the PDP-11/60. Structure factor calculation programs are available for larger DEC computers. Both the diffractometer control programs and calculation programs are written in FORTRAN IV to operate in dynamical priority time-sharing mode.

Crystallographers have looked towards Enraf Nonius with confidence based on our long experience in developing X-ray diffraction equipment, including single crystal diffractometers. The CAD 4 is the most efficient assistant of the modern crystallographer and serves to save his precious time which can be devoted to the real scientific problems now.

Main characteristics of the CAD 4 Kappa Diffractometer

- an absolute minimum of physical obscurities and interferences
- optimal ranges for all angular movements
- full omega rotation without mechanical interferences
- optimal range of reciprocal space can be reached
- azimuth rotation possible for each reflection
- great accessibility of the crystal for all types of accessories
- a most accurate construction due to a bearing mounted axis instead of the arc cradle principle
- optimal long-life accuracy
- easily changed crystal-detector distance
- easy and quick tube alignment with the use of program
- balanced filter unit
- graphite monochromator included in standard package
- continuously variable and program controlled detector aperture
- absolute encoders on all axes and on detector aperture
- 8K or 16K computer with 1.6 Mwords disk standard
- IBM compatible magnetic tape recorder
- most reliable, fast and quiet DECwriter standard terminal
- disk-operating system with very versatile operator's program offers maximum disk utilization and simplified program changes
- most versatile and automatic software
- Polaroid cassette for Laue - or rotating crystal exposures is easily attachable
- low temperature liquid nitrogen or helium systems available
- complete manual control possible. by portable control unit.



Motors and Encoders

All axes are driven with special dc motors with continuously variable slew speeds. This guarantees motion without vibrations even at the highest possible slew speeds.

In going to the next reflection, all axes are driven simultaneously at their maximum speed. The final correct position is always reached with a low speed from the same direction to avoid even the smallest errors due to unavoidable backlash.

Absolute encoders are used. Two encoders are mounted on each axis, a coarse and a fine. This eliminates the difficulties caused by counting encoder revolutions and the dependence upon the computer.

The encoders are of the voltage resolver type. The resolution and electrical angle read out of these encoders is 0.0007°.
Their life is nearly unlimited. The encoders not only achieve this high accuracy but maintain it for a very long time.

A pre-aligned extension piece for the scintillation detector holder to lengthen the crystal-detector distance for protein work is provided with the instrument. The two standard distances are: 173 mm and 368 mm. Other distances are available on request. An extended diffracted beam tunnel which can be flushed by a low absorbing gas is available too.

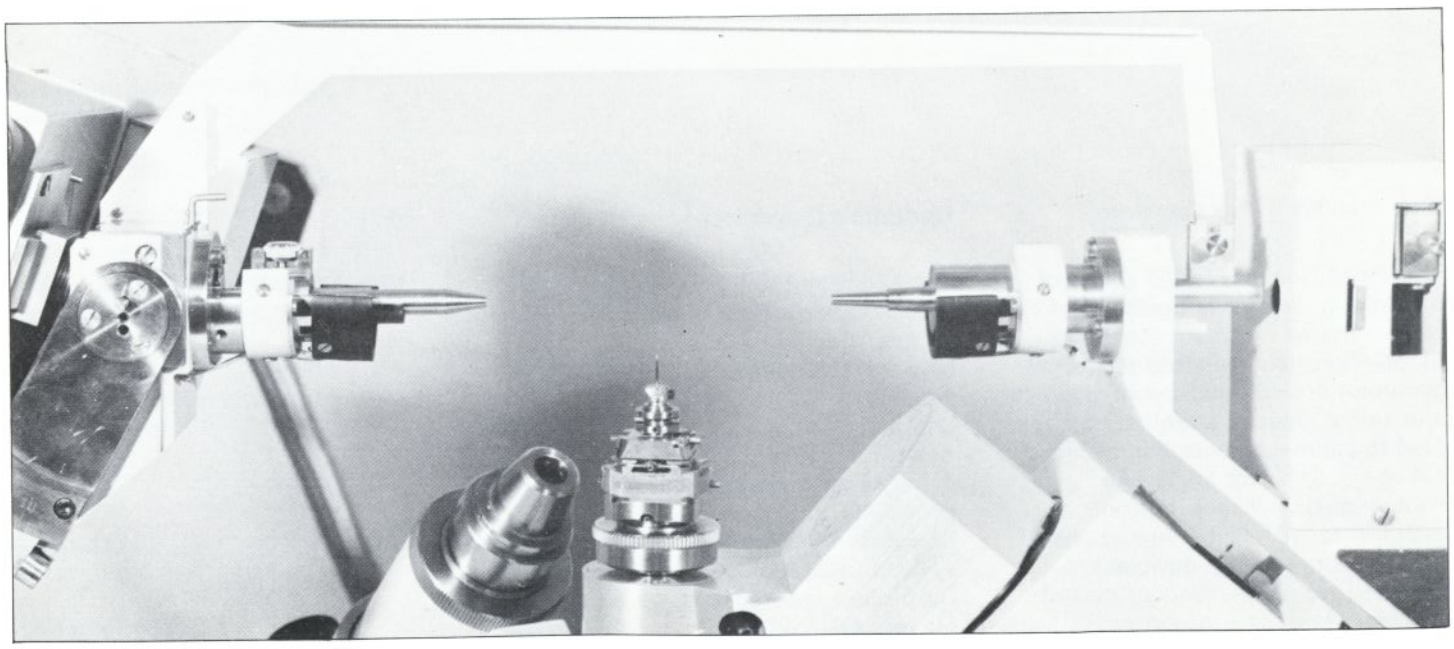
The counter aperture unit has a continuously variable horizontal receiving aperture, a horizontal slit, two diagonal slits and two beam splitters. The apertures are computer controlled but manual control is also possible. The reproducibility of the positions of the different apertures is 10 μ. The height of the aperture is determined bij slits that can be inserted manually (1 mm, 2 mm, 3 mm, 4 mm, 6 mm).

An incident beam balanced filter unit is provided as standard with the instrument and is simply fitted to the tube stand.
One of the filter holders can be positioned and locked in order to balance the two absorption filters. The balanced filters are also programmable or under manual control. In the Enraf Nonius incident beam graphite monochromator system, which is also delivered as a standard with the CAD 4, the monochromator omega axis is transverse to the omega/2 theta axis of the goniometer. Thus the spectral dispersion of the monochromator is parallel to the omega/2 theta axis, resulting in a symmetric geometry for same reflections at positive and negative 2 theta values. A long experience preceeds this monochromator arrangement. The tube shield can be tilted from the equatorial plane to a maximum of 30° while the tube focus adjustments and the take-off angle keep their positions, which is very important for easy adjustment of the total monochromator system. A block containing the omega motion and the graphite monochromator crystal is simply attached to the tube stand and fits in the same way as the balanced filter block. The monochromator crystal can be moved parallel to its omega axis.

Tubeshield, Collimating system and Beamcatcher

The tubeshield is designed to accept fine focus, as well as normal focus tubes. (Philips, AEG, Elliott etc.) It is mounted parallel to the equatorial plane and is an integral part of the goniometer. This assures a rugged construction and makes the goniometer alignment independent of table top movements. x-z translations provide the user with a very accurate focus adjustment device which is easy to handle.
The take-off angle can be adjusted between 0° – 8° with a scale reading for the take-off angle of 0.05°.
The tube filter disk has four positions: Mn, Ni, Zr filters and open. The status of the fail-safe computer controlled X-ray shutter is indicated by two control lights. If both these lights should burn out, the shutter cannot be opened.
The pre-aligned collimator support is mounted on the tube stand.
Collimators rather than inserts are provided. They are available for both fine focus and normal focus tubes.
Standard are:
3 collimators for fine focus tubes and 2 collimators for normal focus tubes.
An extra safety shutter has been built to assure that uncollimated X-rays cannot escape from the system.
A secondary beam tunnel serves to collimate the diffracted beam and to minimize the background. Two different diffracted beam collimators are provided with the instrument.

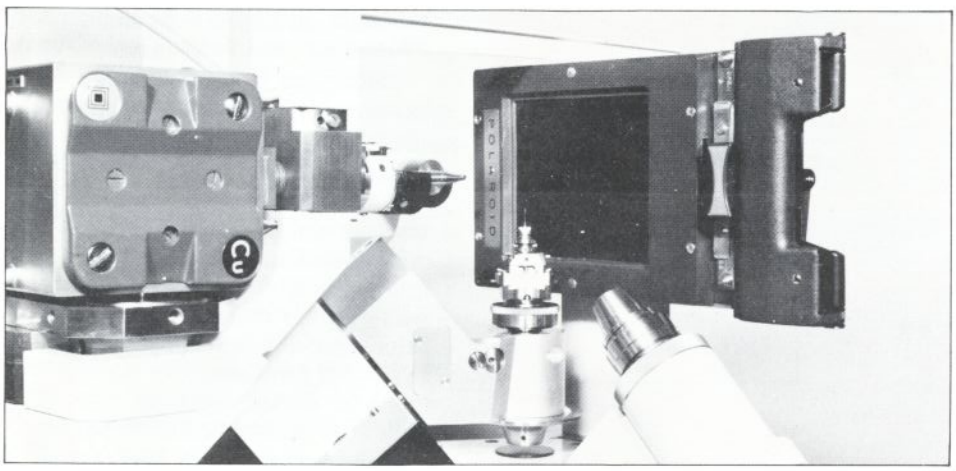
The primary beam catcher is placed to give optimal security and at the same time to allow measurements even at very low 2 theta angles.
Two differently sized beam catchers are provided with the instrument.



A microscope, which gives a comfortable 80 x magnification and has a graduated reticule, is fixed to the omega bracket. This microscope remains aligned during the operation of the diffractometer and does not give any obscuration or physical interference.
It is placed to enable the user of the diffractometer to make the necessary primary centering adjustments of the crystal.
A second microscope which can be mounted independently of any movement of the goniometer is supplied as an extra optional.

A highly stable eucentric goniometer head with a testcrystal mounted is provided as standard with the diffractometer.

All axes, the attenuation filter, the aperture disk, etc., can be controlled with a portable control unit which has in detail the following functions:
- key operated main switch for manual or computer operation of the goniometer
- shutter switch and pilot lamp
- balanced filter switch
- attenuation filter switch
- motor direction switch
- speed range selector
- motor speed continuous variation button
- motor selector for phi, kappa, omega, 2 theta and aperture disk
- collision switch override buttons.



Polaroid Cassette

A Polaroid XR 57/1 cassette can be attached to the goniometer base allowing Laue- or rotating crystal exposures; rotating crystal exposures can be used to calculate a first reciprocal axis matrix of a non-aligned crystal. This provides one alternative to start with a not aligned and possibly unknown crystal. (The other alternative is the very efficient automatic reflection search). The Polaroid exposure will give interesting information about crystal quality and symmetry.

Environmental Enclosure

The new Enraf Nonius environmental enclosure provides maximum safety for the user and at the same time optimum access to the goniometer. The enclosure can be opened from any side. The doors are provided with interlocks closing the X-ray port if one door should be opened during operation.

Computer Hardware

The CAD 4 is available in numerous configurations based upon the user's requirements.

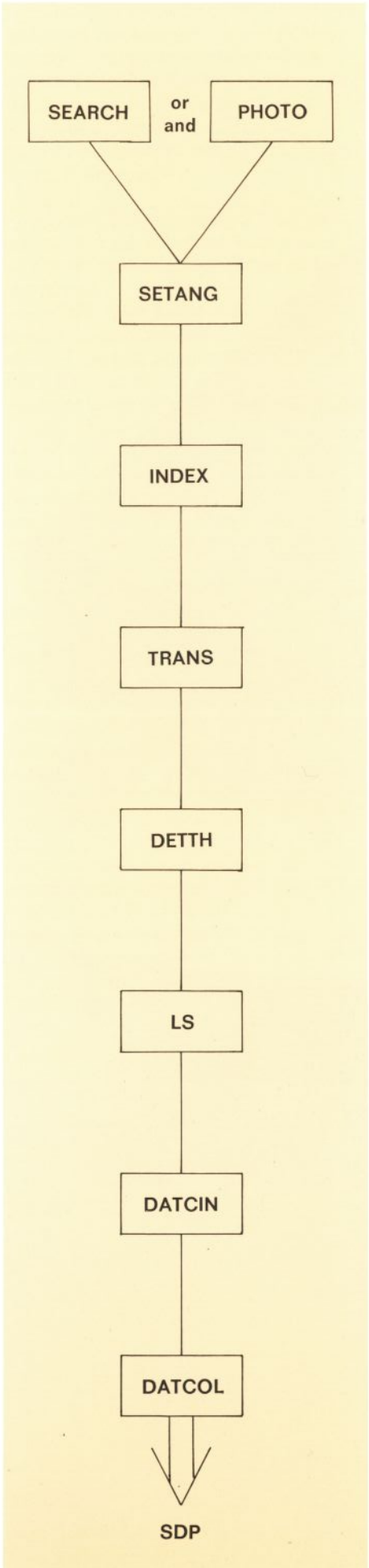
The basic CAD 4 configuration uses the Digital Equipment PDP-8 computer with a 8K core memory together with a movable head disk RK 8. The disk is used to store the programs and the experimental results temporarily. Once read to the disk, all necessary diffractometer programs and mathematical programs remain in the system and can always be used without requiring any special loading.

A highly efficient operator system to communicate via terminal with the computer and to work optimally with the disk is provided with the instrument. The system enables the user to run long programs with a small computer core memory. There is ample space for program extensions.

For systems utilizing an expanded PDP-8 with 16K core memory (CAD 4 F), the programs are supplied in FORTRAN IV. If a PDP-11/34 or PDP-11/60 system is selected for diffractometer control (CAD 4 SDP) FORTRAN IV programs are also used. Then the programs are working in dynamical priority time-sharing mode. If 32K of core memory, floating point hardware, two 1.2 million words disks (or other mass storage disk) and magnetic tape are included, then structure calculating programs are supplied as part of the Enraf Nonius Crystallographic System ENX.
The interface between diffractometer and computer is designed with standard I.C. logic modules, assuring easy interchangeability. The CAD 4 system includes a high efficiency magnetic tape recorder. A synchronous IBM-compatible write/read recorder makes condensed storage of the measuring results possible. Either 7- or 9-track available. Configuration with DEC tape on request. Produced tapes are immediately usable on computers without any conversion, greatly improving data processing.

Standard terminal is the DECwriter. Other terminals are available.

Whilst the software for diffractometer control is equally comprehensive and complete for all versions of the CAD 4 and whilst the operations are the same for CAD 4, CAD 4 F and CAD 4 SDP the language is different. Programs for the basic CAD 4 are written in Assembler, whereas program language for CAD 4 F and CAD 4 SDP is FORTRAN IV.



Condensed CAD 4 Software Description

The 'CAD 4 SYSTEM' is a disk oriented operator system. It is composed of various main programs, subroutines and space for storing required variables. Programs or data are swapped by program from the disk to the core memory. It gives instructions to the operator via terminal or accepts instructions from the operator in a convenient and simple interactive manner. The open structure of the system facilitates changes and the addition of programs to suit particular requirements.

Automatic reflection search program

- works without any input about the crystal to be investigated
- starts with the only optically centered crystal
- leaves the user free to choose the 2θ starting value
- can work on fixed 2θ value if desired, e.g. to find specific reflection
- automatically looks for 25 reflections
- optimizes automatically these reflection positions in 3 angles ω, 2θ, κ
- determines necessary scan width for all reflections found
- determines integrated net intensities of all reflections found
- determines peak position and gravity center of each reflection
- outputs scan profile on terminal on command
- stores a list of the optimized reflection positions with all included information and outputs on request.

Reflection search program using film coordinates input

- starting inputs are the film coordinates of reflections on a rotating crystal Polaroid photograph made on the diffractometer
- calculates automatically correct ω, 2θ and κ values
  looks for correct φ value
- optimizes automatically these reflection positions in 3 angles ω, 2θ, κ
- determines necessary scan width for all reflections found
- determines integrated net intensities of all reflections found
- determines peak position and gravity center of each reflection
- outputs scan profile on DECwriter on command
- stores a list of the optimized reflection positions with all included information and outputs it.

Auto-indexing program

- uses as starting input the previously stored list of 25 or less non-indexed reflections
- investigates the crystal without any further input about the crystal
- determines correct indices for these reflections
- calculates orientation matrix of the crystal
- does least-squares refinement of the matrix components
- calculates metrical tensor (Niggli matrix)
- determines normalized reduced unit cell
- prints pairs of perpendicular directions in direct lattice.

Matrix transformation program

- transforms unit cell and transformation matrix
- transforms orientation matrix
- transforms indices in reflection list on request
- saves transformation on specific file; old unit cell recalled by single command.

Reflection centering programs

- work with an input list of reflection positions, up to 25 reflections are possible
- use program controlled slits for fast running centering program
- produce an accuracy of centering which is better than 0.005° (this program is incorporated in the reflection search program and automatically centers the found reflections)
- prepare a new list of optimized reflection positions which are stored at same location as input list
- use the gravity center for optimization. This avoids $K\alpha_1/K\alpha_2$ split up problem
- contain special program provided to do additional, very accurate determination of 2θ value based on measurements at + and -2θ
- contain special program provided to do scan around any axis or combination of scans with print out of reflection profile and information about reflection centering
- achieve very accurate determination of cell constants and check alignment of the goniometer.

Orientation Matrix programs

- provide different starting input possibilities
  - calculate orientation matrix using unit cell parameters and 2 reflection positions

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"
LS
 1  -1.001   2.000  -2.997   0.0361
 2  -1.001   1.997  -3.998   0.0337
 3  -3.000   1.998  -2.000   0.0239
 4  -1.002   0.999  -4.002   0.0320
 5  -3.001   1.001  -1.999   0.0300
 6  -2.001   0.998  -2.999   0.0377
 7   5.998  -0.002  -0.998   0.0225
 8   0.002   2.006   4.001   0.0700
 9  -0.001   6.000  -0.002   0.0198
10  -0.002  -6.002   0.001   0.0242
11  -0.003   0.998   6.003   0.0328
12   0.003  -1.000  -5.997   0.0271
13   0.000   1.994   8.002   0.0414
RECIPROCAL AXIS MATRIX
   0.082611   0.023330  -0.007429
  -0.052394  -0.035914  -0.091665
  -0.000895   0.077627  -0.001481

NIGGLI-VALUES
   148.1774   163.4779   157.8939
   -33.7179   -55.9565   -49.1129
 VOLUME=      1605.6960

CELL PARAMETERS
    12.1728    12.7858    12.5656
   102.1149   111.4587   108.3945
  -0.209869  -0.365829  -0.315555
"
DIRECT AXIS MATRIX
  11.510980   0.007187  -6.582241
  -0.870474  -0.207156 -10.330611
  -3.862276  12.784171  -2.801172

SIGMA DIRECT AXIS MATRIX
   0.003272   0.001364   0.002561
   0.005523   0.002303   0.004323
   0.003876   0.001616   0.003034

SIGMA CELL PARAMETERS
     0.0034     0.0035     0.0021
     0.0199     0.0206     0.0318
   0.000339   0.000335   0.000527
```

- calculate orientation matrix using a list of at least three reflections
- need no further input if the list formed after optimization or indexing will be used
- do least-squares refinement
- determine accurate unit cell parameters
- determine standard deviation of unit cell parameters and matrix components
- calculate h k l limits
- calculate the vector components
- do all calculations in relation to orientation matrix and goniometer position such as change of azimuth position and recalculation of bisecting position
- correct on command reflection positions automatically in case of focus missettings or optical miscentering of the crystal.

Automatic data collection program

- provides different scan possibilities such as ω , $\theta/2\theta$, and various. ω/θ scan modes which enables user to reduce aperture width to improve peak/background ratios
- does automatic datacollection at various azimuth angles (rotation around diffraction vector) for empirical absorption correction or multiple reflection test
- provides fixed or variable (program control) scan during operation
- uses variable scan angle and receiving aperture width depending on θ ($K\alpha_1/K\alpha_2$ separation)
- generates h k l in a selectable ZIGZAG pattern;
- accepts manual h k l input or prepared list of h k l 's
- skips automatically systematic extinctions
- provides the possibility of Friedel measurements
- provides automatic balanced filter measurements
- does intensity measurement using a continuous scan of 96 single measurements
- stores either: background/intensity/background or complete reflection profile on disk on simple command
- prints short or extended information depending on switch position
- copies short or extended information to tape or other I/0 device, including X-ray exposure time
- outputs to terminal only special data like intensity monitors on request
- provides up to 25 control reflections for intensity monotoring; desired intensity and limits of absolute variation are input values
- measures control reflection after a pre-selected time of X-ray exposure as defined in input
- provides program controlled sequence of data collection,
- reoptimization and recalculation of orientation matrix
- provides angular stability control using up to 25 control reflections
- reoptimization and recalculation of orientation matrix using up to 25 control reflections
- measures all reflections with statistical accuracy defined in input
- calculates scan speed for slow scan after prescan depending on desired accuracy
- skips slow scan depending on given low intensity limit
- does repeated scan, if specified, and integrated intensity measurement, does equality test of the two results
- repeats automatically intensity measurement up to 3 times in case of non-equality
- performs continuous scans around reflections and scans in a certain direction of the reciprocal space
- provides special four circle measuring program to measure intensities at a crystal positioning with minimum absorption loss (important when strongly absorbing materials crystallize in needles or plates)
- modifies in data collection input simply bij changing the concerned address or addresses after interruption, automatic continuation of data collection after a simple command
- saves and recalls all input values
- provides data output to terminal, 7 or 9 track magnetic tape or other selected I/0 device like papertape puncher if requested
- uses standard magnetic tape code BCD, ASCII, EBCDIC or any other available code
- data blocks written on magnetic tape are checked after writing and rewritten if wrong; all blocks are written twice
- calls for operator in case high voltage is off, magnetic tape at end or similar situations.

General operator program

- does calculations from Eulerian geometry to Kappa geometry and vice versa
- replaces manual operation with the diffractometer by terminal commands such as
  - positioning to angles given in Kappa or Eulerian geometry
  - positioning to h k l
  - positioning to different azimuth angle
  - positioning to negative 2θ value
  - positioning to Friedel - correspondent reflection
  - display of precise encoder position
  - operation of shutter, aperture, diaphragm and filter
- does ω , 2θ or χ (combination of κ and ω) scan or any other combination scan at a given position using input value for scan angles, scan speed, and aperture width
- displays reflection profile on terminal
- outputs intensity and deviation of peak position and center of gravity.

Loading and saving programs

- program to load disk with software from magnetic tape
- program to save disk contents on magnetic tape
- program to make terminal display of the tape contents.

Diagnostic programs

- program to check operation of magnetic tape
- program to check the interface
- program to check the goniometer function including encoder system, motors, friction, etc.

Data reduction program

- does Lorentz-polarisation correction
- does empirical absorption correction
- works on datacollection output file
- produces new disk file with observed structure factors.

Other programs

The user is supplied with DEC standard programs.
The complete package exceeds all software of comparable computer systems. Some of these standard programs are stored on the disk together with Enraf Nonius programs.

CAD 4 SDP

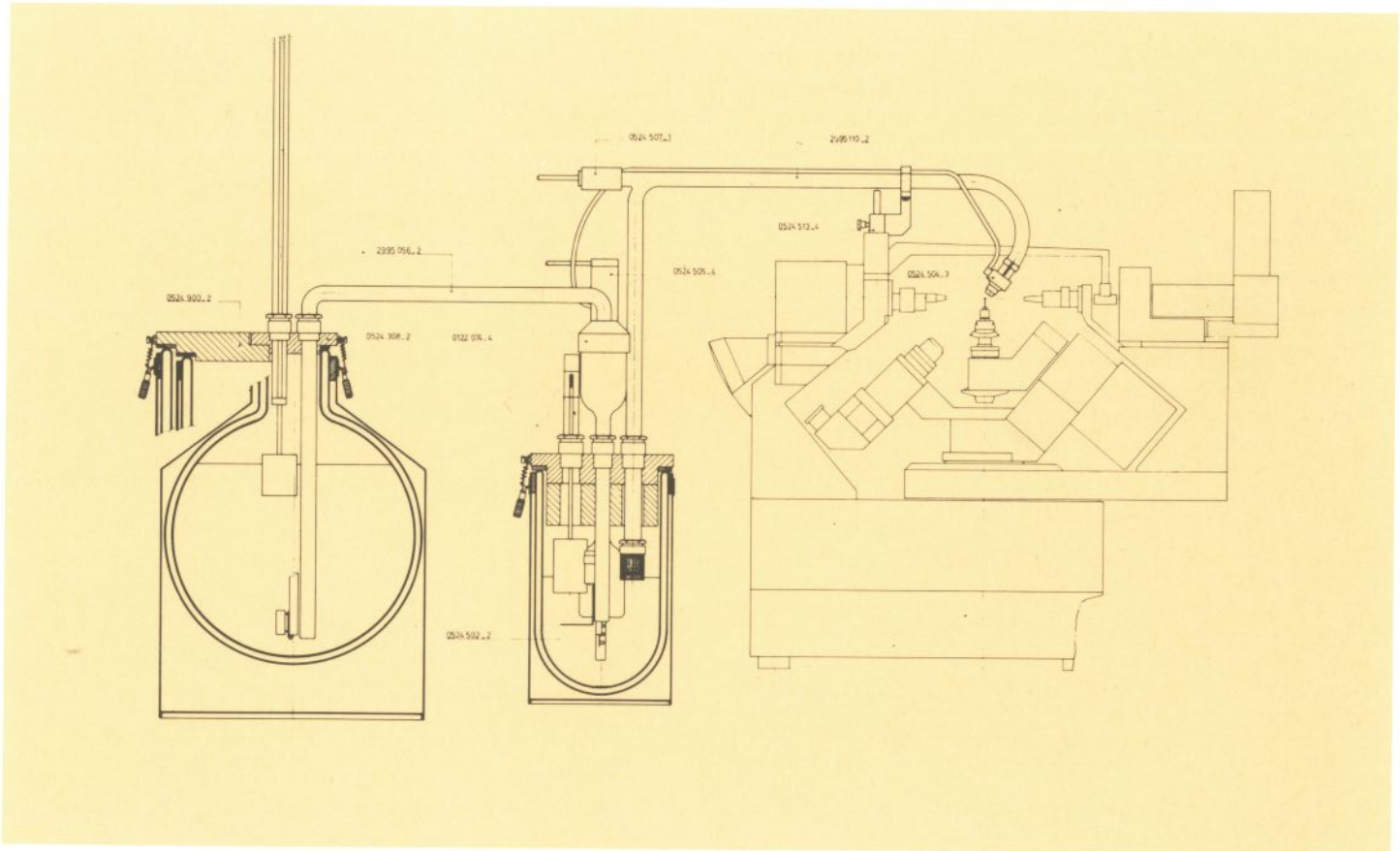
The growing concern about the cost of crystallographic computing has encouraged crystallographers to look for alternative ways of performing their calculations. With the advent of relatively inexpensive small computers, capable of rapid execution times and readily accessible disk storage, it became clear that a powerful new crystallographic tool was potentially available. The need for an integrated library of crystallographic programs designed for a small computer was apparent.

The Enraf Nonius **structure determination package** (SDP) is such an integrated set of remarkably fast programs, developed for use on the PDP-11 series of computers.
The complete Enraf Nonius structure determination system CAD 4 SDP consists of the standard X-ray generator, Kappa-axis-goniometer hardware and counting chain hooked up to a PDP 11/34 of PDP 11/60 computer system. The computer hardware includes a minimum of 32K core memory, floating point hardware, two moveable head disk systems, IBM compatible tape drive, line printer/plotter and one or more terminals.
CAD 4 diffractometer and structure calculations SDP are run simultaneously in a time share mode program.

Language for both diffractometer control and structure calculations is Fortran IV with the exception of a few assembler language subroutines.
The SDP programs compile, link, and execute in 32K of core and take advantage of rapid access to a moving head disk for storage of large arrays.

The package includes programs for data reduction, direct methods phase determination, Fourier calculations, least-squares refinement, function and error calculations, plot and table productions, etc. For more detailed information is referred to the CAD 4 SDP leaflet.

Low temperature work



The Enraf Nonius Universal Low Temperature Device, FR 524H, uses liquid nitrogen cooling for temperatures down to approximately –170°C. It does not limit the accessible diffraction sphere and the movements of the goniometer in any way.
Permanent improvements of this accessory achieved in close co-operation with the Institute for Structure Chemistry of the University of Groningen have led to an easily operable system with good temperature stability and a low temperature gradient at the crystal position.

A cryostat to work at temperatures down to approximately 10° K has been developed for use with the CAD 4. This cryostat, CT 73, uses liquid helium. Whilst there is some obscuration caused by the device, it leaves great directional freedom under these extreme circumstances.
For more detailed information read the specific literature.

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