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The Oslo Electron Diffraction Units for Gas Work

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A new electron diffraction sector apparatus, primarily intended for gas work and now in use for some time is described. The high tension source, electron gun and magnetic lens are of commercial type, the rest of the apparatus has been designed and constructed in our institute. The new apparatus has made it possible to increase the accuracy of the intensity measurements and to extend the intensity measurements far beyond the s-range earlier investigated. Besides the advantages thus opened with respect to precise measurements of molecular parameters it has become possible to increase very markedly the resolution power of the procedure.

The use of a rotating sector to compensate for the steep fall in the background of electron diffraction diagrams from a gas was suggested independently by Trendelenburg[1], Finbak[2] and P. P. Debye[3] in the thirties. In Oslo, the method has been in use since 1940, and the advantage of the method as compared to the visual method has since then been fully realized. A series of the problems studied in our laboratory, particularly those involving the difference procedure, would in our opinion hardly have been solved with the use of the visual method[4–15].

The first sector electron diffraction apparatus in Oslo was designed by Chr. Finbak in 1936. As this apparatus has earlier been described[16,17], and since the main principles are very much the same as for that recently designed by Brockway and Bartell[18], it will only be mentioned briefly in the present report.

The usefulness of the old apparatus may perhaps most easily be demonstrated by reproducing a photometer curve of a diagram of a comparatively large molecule. In Fig. 1 is presented, therefore, a photometer curve of *sym.* triphenylbenzene.

One of the main differences between this apparatus and that of Brockway and Bartell is the mounting of the sector. A central axle is applied in the Oslo apparatus instead of the peripherally mounted ball-bearing system. We have found the central axle simple and convenient and have, therefore, applied this type of construction also in the new apparatus. The electron source of the old

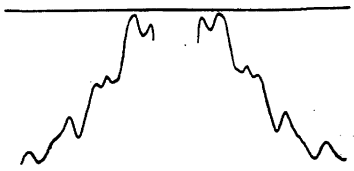

Fig. 1. Photometer record of sym. *triphenylbenzene from a diagram taken in the old sector apparatus.*

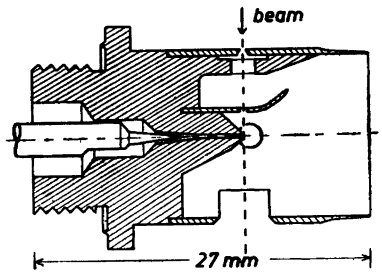

Fig. 2. Gas nozzle with needle valve used in the old apparatus (simplified).

apparatus consists of a cold-cathode discharge tube. The high voltage supply is also an old-fashioned construction but gives a stable direct current.

In order to secure the best possible construction for the new apparatus many of the details were first tried out in the old unit. For instance, a whole series of different gas-nozzle designs were tried out. A nozzle type which has been much used is illustrated in Fig. 2. Various liquid-air traps have been tried and also different sector shapes and sector screening factors. Most of the diagrams of the old apparatus were taken with an s^2 or s^3 sector.

DESCRIPTION OF THE NEW OSLO APPARATUS

The new apparatus was designed in collaboration with Professor Chr. Finbak (†). The ideas leading to many of the improvements we owe to him. The apparatus has to a great extent been built in the workshop of our institute. Exceptions are the high voltage supply and the electron gun which were purchased from Metropolitan-Vickers, England. This is standard equipment for an electron microscope. Only one magnetic lens is used for focussing the beam.

Fig. 3 is a simplified sketch and Fig. 4 a photograph of the apparatus. As may be seen from the scale, the dimensions are larger than for previously-built diffraction units. We chose this size in order to be able to place various kinds of auxiliary equipment inside the vacuum system and to avoid limitation in scattering angles due to the geometry of the apparatus. The various parts of the apparatus are fitted together with O-rings. O-rings are also used for the windows and the plate-box door.

The vacuum chamber consists of two steel cylinders, the inner diameters of which are 50 and 63 cm, respectively. The total inner height is 80 cm and the volume approximately 180 liters. The inner edge of the (shelf-like) connection between the cylinders is supported by steel pillars.

The main opening giving access to the chamber is a door (30×13 cm^2) in the bottom cylinder. Through this door the plate box may be inserted when the carriage is in its lowest position. In the top and bottom plates of the chamber and in the side walls of the upper cylinder a series of openings is present. Thus, one may introduce electron gun, pumps, gauges, cooling trap and various

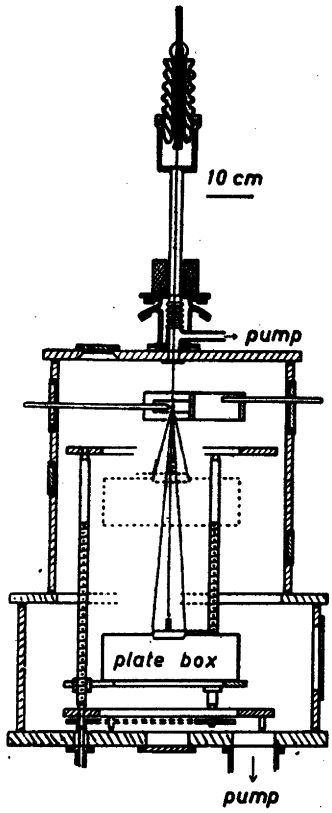

Fig. 3. Simplified section sketch of the new diffraction apparatus.

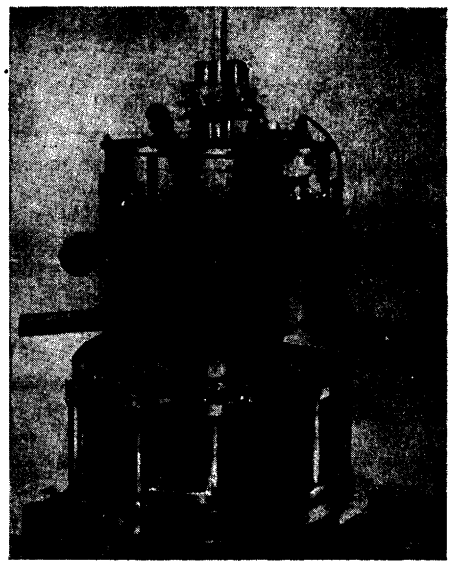

Fig. 4. Photograph of the main part of the new apparatus.

auxiliary equipment. Plexiglas windows are provided. Four standard sizes for the openings have been chosen, thereby allowing the same pieces of equipment to be moved from one position to another. This, of course, adds to the flexibility of the whole arrangement.

The plate box. The capacity of the plate box (Fig. 5) is 15 plates or more of maximum size 6 × 24 cm². The box is mounted on tracks and the loading or disloading is therefore very easy. The unexposed plates are stored in the first of three compartments. By means of a chain driven system operated by one single control, the plates may be transferred to the second compartment for exposure, and after exposure to the third compartment. The same control also opens and closes the exposure lid. In the exposure compartment, the two ends of the plates are pressed upwards against slide rails in the top of the box, the emulsion side thus being brought in a well-defined position. The plate-box mounting can be moved up and down and the distance between the photographic emulsion and the diffraction point thus be continuously varied. In order to keep the plate box exactly horizontal during this movement, the lift is

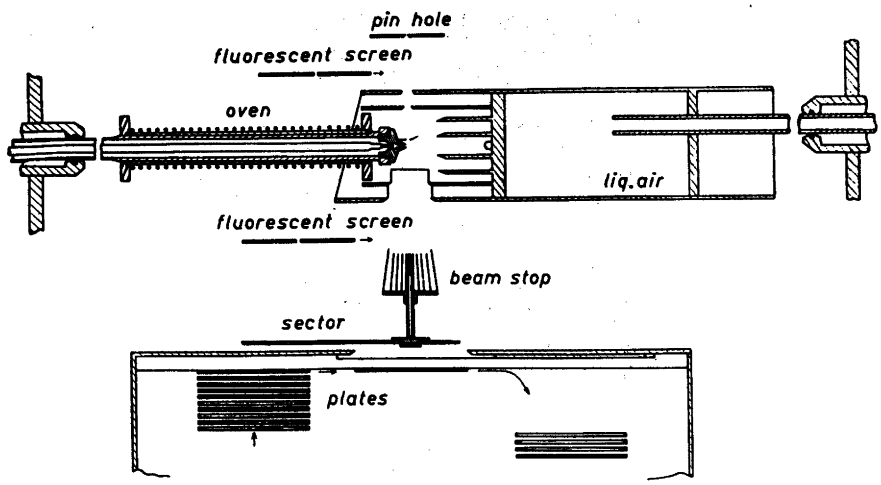

Fig. 5. Section through oven, liquid-air trap, and plate box with sector and beam stop (simplified).

operated by three screws which are rotated simultaneously by a chain connecting the toothed wheels at the end of each screw. The lift can be operated from the outside of the apparatus by means of one of the vacuum-tight shaft-bearings. By simply choosing the proper windows for nozzle and the liquid-air trap it is possible to vary the distance between the photographic plate and the diffraction point from a few centimeters to about half a meter.

The sector should be kept in a position close to the photographic plate although the nozzle-to-plate distance can be changed within wide limits. The sector mount, therefore, forms part of the carriage system. If necessary, however, it may be unfastened by a single hand operation. Gear transmissions, intended for rotation of the sector, as well as for adjustment in two horisontal directions, have been provided. If desired, the sector may also be moved out of the plate area. The carriage movements do not interfere with the operation of the sector or the plate box.

The sector found to be most satisfactory at the present time has a spiral form as shown in Fig. 6. Within the greater part of the s-interval the screening factor is approximately proportional to s^3; in the inner part, the opening is made larger than corresponds to this function. The sector is made from a phosphorus-bronze sheet of thickness 0.2 mm. To avoid deviation from planarity the sector is supported by duraluminium edges on both sides. The sector is operated by an electric motor placed outside the apparatus. The shape of the sector has been carefully studied both by direct measurement under the microscope and by diffraction experiments. From these studies, sector correction curves for use in our structure-determination work have been computed. The beam stop, mounted on the top of the sector, consists of a series of concentric cylinders. The beam stop used for the short-distance pictures, where we are mainly interested in the largest s-values, consists of seven cylin-

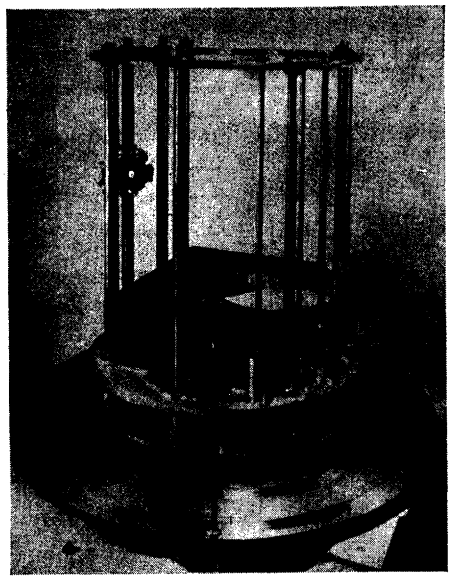

Fig. 6. Photograph showing the carriage arrangement. Sector replaced by a paper model.

ders. In the bottom of the beam stop is placed a layer of carbon black, which has a very low secondary electron emission. Colloidal graphite suspension can be used for coating the cylinder walls. The use of an effective beam stop seems to be of the greatest importance in order to obtain reliable intensity data at large s-values.

The nozzle and liquid-air trap designs seem also to be of great importance for the quality of the diffraction diagrams. Several oven- and nozzle types for various purposes have been constructed. The nozzle and the trap are placed on opposite sides of the apparatus, but at the same height. The end of the trap is a cylinder surrounding the nozzle. In this way, practically all the gas leaving the nozzle is condensed in the first trap and is accordingly prevented from reaching the rest of the apparatus. In Fig. 5, a typical nozzle-trap combination is shown.

The new apparatus has enabled us to enlarge our s-interval considerably. In the old apparatus we were able to obtain intensity values ranging from $s = 3$ Å^{-1} to nearly $s = 30$ Å^{-1}. The new apparatus gives intensity values extending from s equal to 1 Å^{-1} to s-values of about 60 Å^{-1}. Experimental curves giving intensity values over a large s-range are reproduced in Fig. 7. Corresponding theoretical curves are also given, for the sake of comparison. In our standard procedure we usually limit ourselves to three different distances between diffraction point and photographic plate, *viz.* approximately 49 cm, 19 cm and 12 cm. The experimental intensity curves like those reproduced in Fig. 7 are obtained in the usual way: A Leeds and Northrup microphotometer is used, and the plates are oscillated during the process of recording. The

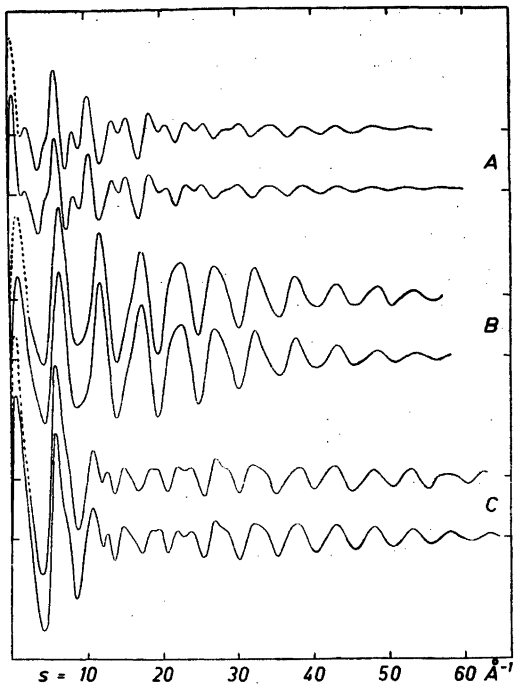

Fig. 7. Experimental and theoretical intensity curves of cyclooctatetraene *(A), NO_2 (B), and vinylacetylene (C).*

photometer curves are read off in a logarithmic scale and the blackness is transferred into intensity values before the theoretical background is subtracted.

The wavelength control is based upon diffraction pictures from gold foils or from evaporated gold films. Other reference substances have been tried, but at present we feel that gold gives the most reproducible values. An *s*-sector is used when taking calibration pictures. Without a sector, a slight decrease in wavelength with increasing *s*-values is observed. This decrease is probably due to electric charging effects in the photographic plates.

As we have found our present commercial high-voltage meter not to be sufficiently accurate for precision determinations we are building a new instrument for high voltage control. This outfit has, however, not yet been in operation and it will not be described here.

The apparatus has been briefly described by one of us in a lecture given at the Eighth Scandinavian Chemistry Congress in Oslo, June 1953 [19].

We want to express our sincere gratitude towards *Norges Teknisk-Naturvitenskapelige Forskningsråd* and *Norges almenvitenskapelige forskningsråd* for grants enabling us to build the new unit and use it for structure determinations. We are also very much indebted to Mr. S. Sørensen, senior instrument maker in our institute, for his excellent contribution to the construction work, and to our colleague cand. real. A. Almenningen

for his most valuable help in setting up the apparatus and his initiative and skill during the running-in period. Very helpful suggestions regarding technical improvements are due to him. We also want to express our gratitude to instrument maker Th. Nilsen, Trondheim, for having taken care of the construction of the plate box.

REFERENCES

1. Trendelenburg, F. *Wiss. Veröffentl. Siemens-Konzern* **13** (1933) 48.
2. Finbak, Chr. *Avhandl. Norske Videnskaps-Akad. Oslo I. Mat.-Naturv. Kl.* **13** (1937).
3. Debye, P. P. *Physik. Z.* **40** (1939) 404.
4. Hassel, O. and Viervoll, H. *Tidsskr. Kjemi, Bergvesen Met.* **3** (1943) 32.
5. Bastiansen, O. and Hassel, O. *Nature* **157** (1946) 765.
6. Bastiansen, O. and Hassel, O. *Tidsskr. Kjemi, Bergvesen Met.* **6** (1946) 70.
7. Bastiansen, O. and Hassel, O. *Tidsskr. Kjemi, Bergvesen Met.* **6** (1946) 96.
8. Bastiansen, O. and Hassel, O. *Acta Chem. Scand.* **1** (1947) 489.
9. Bastiansen, O., Ellefsen, Q. and Hassel, O. *Research (London)* **2** (1949) 248.
10. Bastiansen, O. *Acta Chem. Scand.* **3** (1949) 408.
11. Bastiansen, O. *Acta Chem. Scand.* **3** (1949) 415.
12. Bastiansen, O., Ellefsen, Ø. and Hassel, O. *Acta Chem. Scand.* **3** (1949) 918.
13. Bastiansen, O. *Acta Chem. Scand.* **4** (1950) 926.
14. Bastiansen, O. *Acta Chem. Scand.* **6** (1952) 205.
15. Bastiansen, O. *Acta Chem. Scand.* **6** (1952) 875.
16. Finbak, Chr., Hassel, O. and Ottar, B. *Arch. Math. Naturvidenskap* **44** (1941) No. 13.
17. Hassel, O. and Viervoll, H. *Acta Chem. Scand.* **1** (1947) 149.
18. Brockway, L. O. and Bartell, L. S. *Rev. Sci. Instr.* **25** (1954) 569.
19. Bastiansen, O. *Det 8. Nordiske Kjemikermøte. Beretning og Foredrag.* **1954** 139.

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