extremely low frequencies; the resulting form (Fig. 2, 12%) displays an upward curvature. Thus the shape of the diagram indicates the relative importance of the two polarisation mechanisms proposed. At high humidities (Fig. 2, 74%) a fully developed circular arc appears. The possible contribution of variation in n to this situation is not yet evaluated.

M. Shahidi
J. B. Hasted
A. K. Jonscher

Birkbeck College and Chelsea College, University of London, UK

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Optical resolution by chromatography at low temperature

We report here the first successful optical resolution experiments using low temperature chromatography, for which we used the tris-acetylacetonato (acac) complexes of Al(III) and Fe(III). Since the apparently unsuccessful attempts to resolve Fe(oxalate)$^{3-}$ at -30 °C on starch[1], only room temperature experiments have been reported (for example, refs 2–4).

Using a high-pressure pump (20 MPa) the eluent 4:1 isopentane–ethyl ether, was used here, but 2 chlorobutane (or dimethyl ether could be used down to 140 K) was run (0.5–2 ml m^{-1}) through the column (diameter 7.5 mm, length 300 mm) enclosed in a themostated Cu-block in a liquid N_2 cryostat. The sample (typically 0.05 ml of a saturated ethanol solution) was injected through a septum into the eluent line just outside the cryostat. The eluate was led from the column through an optical cell and then out of the cryostat. All optical windows were birefringence free.

The column filling was D-lactose and Al_2O_3: a saturated solution of 4.5 g D-lactose was stirred with 10 g chromatographic Al_2O_3 (basic, 100–150 mesh). About 50% of the water was evaporated at 80 °C, and the slurry obtained was mixed with 100 ml benzene. The liquid phase was removed with a glass filter, and the filtrate was dried with air at 70 °C. Aggregation of the Al_2O_3 particles thus seemed to have been prevented.

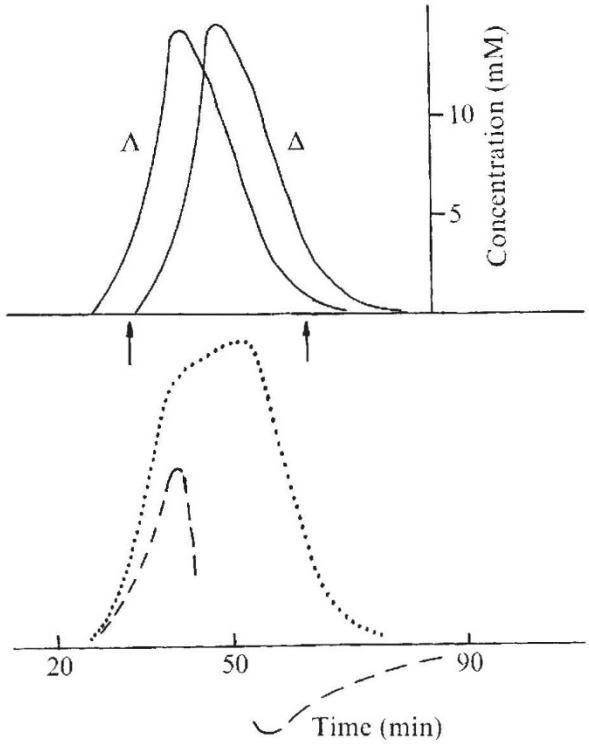

Fig. 1 A typical eluation pattern,) Al(acac)$_3$. Absorbance (···) and circular dichroism (– – –) at 303 nm against the retention time and possible corresponding concentrations of the enantiomers.

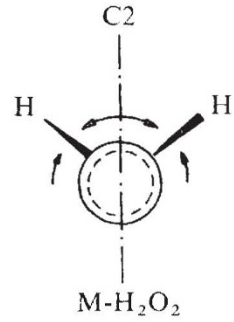

Fig. 2 The M–H_2O_2 molecule.

A high optical yield was suggested by an almost constant ratio between the circular dichroism (CD) and the absorbance (A) of the early fractions after the breakthrough and in some cases also by a shoulder on the absorbance curve. Fe(acac)$_3$ and Al(acac)$_3$ could be resolved below -100 °C and -30 °C, respectively. By analogy with other M(acac)$_3$ complexes on D-lactose, it is concluded that the enantiomer with the absolute configuration Λ (IUPAC nomenclature[5]) appears first from the column. The observed CD of the Fe complex was fairly diffuse (extremum at about 270 nm: $CD/A = +1.4\times10^{-4}$) while Al(acac)$_3$ showed the two expected bands at 300 nm due to dipole couplings between locally excited transitions in the acac ligands: $\Delta\varepsilon/M^{-1}\ cm^{-1}$ (the wavelength of extremum in nm, assuming perfect optical purity, is in parentheses): -54 (276), $+115$ (303). The spectral details and the inversion kinetics of Al(acac)$_3$ are to be discussed elsewhere[6].

The use of optical activity methods would be vastly expanded if molecules could be resolved which are (statistically) inactive at room temperature but have asymmetric ground state configurations. Hydrogen peroxide (H_2O_2), for example, with O–O axis rotation barriers which are symmetric about the C_2–O–O plane (see Fig. 2), should be a mixture of equal amounts of the two optical isomers M and P (for nomenclature see ref. 7) at low temperatures. Preliminary studies on its analogue S_2I_2 indicate that it may be resolved at 77 K by photolysis by circularly polarised radiation, as has been shown already for 1,2-dithiane[8]. With only four atoms this is the simplest conceivable optically active species.

Ivan Jonas
Bengt Norden

Inorganic Chemistry, University of Lund Chemical Center, Box 740 S-200 07 Lund 7, Sweden

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