

the cell for containing the solution. The experiment was carried out with a 1-cm. layer of caryophyllene nitrosite solution (1 per cent in benzene), the filter being adjusted to transmit light in the region of maximum absorption ($\lambda = 6750 \text{ \AA}$). First, the $\lambda/4$ plate was oriented to give left-handed light, and the indicator on the galvanometer scale was set to 35 cm. The $\lambda/4$ plate was then rotated through 90° (to give right-handed light) and the indicator was seen to move back about 5 cm. When a Zeiss-Winkel monochromator was used instead of the Christiansen filter, satisfactory readings were obtained only in the neighbourhood of 6000 \AA ., so that greater over-all sensitivity was required. This is available in the Unicam spectrophotometer, and the instrument can be readily adapted for investigating circular dichroism.

The only additional apparatus required is an attachment (for producing circularly polarized light) which contains a Glan-Thompson prism and a $\lambda/4$ plate, the latter being selected for the required region of the spectrum. In the standard model of the Unicam instrument, there is a tubular opening (which contains a quartz lens) between the cell compartment and the exit slit of the monochromator. A brass tube (for holding the Glan-Thompson prism) is inserted behind the lens, and sufficient of the tube projects to provide a bearing on which the $\lambda/4$ plate holder can rotate. A metal plate (shaped to fit into the front of the cell compartment) is soldered to the tube to hold it in position and to carry stops for limiting the rotation of the $\lambda/4$ plate holder between $+45^\circ$ and -45° from the vertical. The optical components are then inserted so that, when the $\lambda/4$ plate is in the $+45^\circ$ position, right-handed light is transmitted. It is also necessary to reduce the length of the cell carrier by about 1.5 cm.

Circular dichroism ($\epsilon_l - \epsilon_r$) is measured with the instrument in the following way. (1) Place the cell containing solution in the light beam. (2) Adjust the slit-width to give 100 per cent transmission (density = 0) for right-handed light. (3) Rotate the $\lambda/4$ plate through 90° . (4) Take the reading on the density scale. This value divided by lc gives $\epsilon_l - \epsilon_r$. If this should be negative, then the 100 per cent transmission setting is made with left-handed light.

Afterwards, a lever arrangement was added for rotating the $\lambda/4$ plate without opening the cell compartment, but this refinement is not essential.

When circular dichroism is obtained from ellipticity measurements (visual or photographic), accuracy is limited by the necessity of using quite dilute solutions. With the photo-electric method, much more concentrated solutions can be employed. A 1 per cent benzene solution of caryophyllene nitrosite in a 2-cm. cell showed a difference in transmission of 30 per cent when left-handed light ($\lambda = 6750$) was changed to right-handed. This result gives some idea of the magnitudes involved. The method has also the advantages of speed and simplicity of operation.

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¹ Fresnel, "Oeuvres complètes", 1, 719 (Paris, 1866).

² Cotton, *Ann. Chim. Phys.*, 8, 347 (1895).

³ Bruhat, *Ann. Physique*, 3, 232 (1915).

⁴ Kuhn, *Trans. Farad. Soc.*, 28, 293 (1930).

⁵ See Mitchell, "The Cotton Effect" (London, 1933).

⁶ See Lowry, "Optical Rotatory Power" (London, 1935).

⁷ Mitchell and Cameron, *J. Chem. Soc.*, 1965 (1938).

Spectrographic Determination of a Major Constituent of a Solution

C. FELDMAN^{1,2} has recently described a solution spark technique, wherein the solution under investigation is carried in an upper electrode consisting of a narrow graphite cup which has a porous base permeable to the solution, the lower electrode being a solid graphite rod.

The possibilities of adapting this technique for use with apparatus commonly available in Great Britain have been under examination by us, the aim being the determination of a major constituent—zinc—in solutions containing complementary amounts of copper and zinc.

Using the Hilger medium spectrograph and standard spark source, an analytical curve has been obtained from the line-pair Zn 2502.001/Cu 2544.802 for the range 10–90 per cent zinc, 90–10 per cent copper. A reproducibility test on a sample of Naval brass showed: average of 32 determinations, 36.1 per cent zinc; highest figure, 37.5 per cent, lowest figure, 35.0 per cent; standard deviation, 0.71 per cent—in terms of zinc content, 1.97 per cent.

On low-alloy brasses and solutions of copper and zinc over the range 25–45 per cent zinc, the accuracy of single determinations has been better than ± 2.0 per cent for the seventy-eight results so far obtained. 3.0 mgm. of brass suffices for an analysis in duplicate.

These results would seem to commend the method as having wide potentialities for extending the range of spectrochemical analysis.

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¹ *Anal. Chem.*, 21, 1041 (1949).

² *Anal. Chem.*, 21, 1211 (1949).

Crystal Structure of Ammonium Bicarbonate and a Possible Relationship with Ammonium Hypophosphate

PRELIMINARY work on the structure of ammonium bicarbonate was done by Mooney¹ in 1932, who reported that the space group was *Pccn*.

We have recently found the positions of the atoms in the structure. Approximate atomic co-ordinates, which may yet be modified slightly as a result of work which is in progress, are given below expressed as fractions of the unit-cell edges taking a centre of symmetry as the origin.

	<i>x</i>	<i>y</i>	<i>z</i>
C	0.0	0.24	0.16
O	-0.03	0.19	0.03
O	-0.03	0.19	0.28
O	0.06	0.35	0.17
N	0.25	0.01	0.41

These co-ordinates are consistent with the presence of planar CO₃ groups of the usual dimensions lying on either side of the diad axes with their planes inclined slightly to the *bc* plane of the unit cell. The CO₃ groups are joined in strings parallel to [*c*] by hydrogen bonds forming, as it were, polymeric anions. This is exactly similar to the hydrogen-bonded strings of CO₃ groups found by Zachariassen² in sodium bicarbonate; in other respects the structures of sodium and ammonium bicarbonates show minor differences.