

Extension of the grating range of the SP100 spectrometer

This content has been downloaded from IOPscience. Please scroll down to see the full text.

1965 J. Sci. Instrum. 42 352

(http://iopscience.iop.org/0950-7671/42/5/420)

View the table of contents for this issue, or go to the journal homepage for more

Download details:

IP Address: 142.58.12.80 This content was downloaded on 21/06/2017 at 00:23

Please note that terms and conditions apply.

You may also be interested in:

A simple, continuous level indicator for cryogenic liquids T Ashworth

A jacketed cell for the Bendix NPL polarimeter L K Dalton

A simple specular reflectance attachment for infra-red spectrometers D S Allam

A simple differential pressure system for measuring depths of cryogenic liquids W L Pope and E F McLaughlin

A simple semi-automatic cryostat to operate at temperatures above -196°C A Grütter and J C Shorrock

 $\frac{Cryostat \text{ for use in the temperature range 150-250° K}}{J J Lawrance and W I Stuart}$ 

An airborne infra-red solar spectrometer

J T Houghton and J P Chamberlain

Infrared spectroscopy of adsorbed layers. II. Investigation of calorimetric infrared spectroscopy H Gentsch and J C Snaith

A thermoelectrically activated instrument for the determination of levels of cryogenic liquids T Ashworth and H Steeple

liquid contained in any type of vessel. During the course of this work it has become evident that a thermocouple can be used very simply and effectively to act as a continuous level indicator for liquids contained in Dewars with parallel, lowconducting walls.

Operation is based on the dependence of the temperature  $T_L$  of a point P on the inside wall of a Dewar vessel (at a distance L from the top) upon the distance l of the liquid surface below this point. The temperature  $T_L$  is also dependent upon the value of L and the temperature  $T_0$  at the top of the Dewar, which is a function of the ambient temperature.

Thus for a given Dewar vessel containing a particular cryogenic liquid,

$$T_L = f(l, L, T_0).$$

Although this function is difficult to calculate with any great accuracy, it is sufficient for the present requirements that observation and approximate theory show  $T_L$  to be a monotonic increasing function of l, for l > 0. Thus by attaching one junction of a thermocouple to the point P and placing the other junction at the bottom of the Dewar a thermoelectric voltage is developed when l > 0; the magnitude of the voltage is also a monotonic increasing function of l.

Typical calibration curves for glass and Monel Dewars of the signal from a copper-constantan thermocouple against level of nitrogen remaining are shown in the figure. A 'Spot' galvanometer (Cambridge Instrument Co., type 41154, 20  $\Omega$ , 19 mm  $\mu$ A<sup>-1</sup>) was used as indicating instrument. The scatter of points from a smooth curve for any particular run was approximately 0.01 cm; from run to run the variations of level for a given signal were less than 0.1 cm, these being attributed to changes of ambient temperature. Overall the level could be found to a certainty of better than  $\pm 0.1$  cm.

On refilling to some level for which l > 0, the time taken for the temperature to reach the equilibrium values was about 10 minutes. As one would expect, the level indication obtained before such time had elapsed did not follow the calibration curve. The shape of the calibration curve for low levels of nitrogen in the glass Dewar is caused by the size of the sinker used.

Although glass Dewars for helium were not available, a simple calculation shows that for use with helium a Fe/Au-Chromel thermocouple (Berman, Brock and Huntley 1964) would produce ample signal to activate a 'Spot' galvanometer. The influence of ambient temperature variations is expected to be less than with liquid nitrogen because of the lower temperatures involved.



Typical calibration curves of level against signal for use with liquid nitrogen. A, curve when used in glass Dewar; B, curve when used in a monel Dewar. Inset: arrangement of element in a glass Dewar.

Although the use of a thermocouple as described above is extremely simple, it does provide a reliable, continuously indicating, level-sensing element capable of an accuracy well suited to the requirements in many research cryostats. The slow recovery time on filling to an intermediate level is not detrimental since the upper junction could be placed at the point to which filling is required; the response to zero signal is then immediate and the subsequent indications arereliable.

#### References

- ASHWORTH, T., and STEEPLE, H., 1964, J. Sci. Instrum., 41, 782.
- BERMAN, R., BROCK, J. C. F., and HUNTLEY, D. J., 1964, Cryogenics, 4, 233.

J. SCI. INSTRUM., 1965, VOL. 42

## Extension of the grating range of the SP100 spectrometer

## J. F. OGILVIE

Department of Physical Chemistry, Lensfield Road, Cambridge MS. received 11th January 1965

Abstract. A modification to the Unicam SP100 infra-red spectrophotometer has been devised to enable the grating range of the instrument to be extended beyond  $4500 \text{ cm}^{-1}$ .

Most commercially available grating infra-red spectrometers have 4000 cm<sup>-1</sup> as the upper frequency limit of the scanning range. Near or beyond the  $3650 \text{ cm}^{-1}$  end of the second grating range of the Unicam SP100 spectrophotometer lie the hydroxyl stretching vibrations of water and many alcohols both in the vapour phase and in dilute solution in non-polar solvents (Pimentel and McClellan 1960). The object of this note is to explain how the SP100 spectrometer can be easily modified to increase its grating range.

In the standard SP100 spectrometer (Tarbet and Daly

1959) with an SP130 grating accessory, there are two gratings: 1950 lines per inch blazed at  $1250 \text{ cm}^{-1}$  and used between 650 and  $2150 \text{ cm}^{-1}$ , and 3000 lines per inch blazed at 3000 cm<sup>-1</sup> in the second order for use from 2150 to  $3650 \text{ cm}^{-1}$ . Both gratings are employed in series with a sodium chloride prism to separate the orders and also to contribute to the overall dispersion; the NaCl prism is also used alone as a single monochromator of small dispersion for the range 2000-8000 cm<sup>-1</sup>. Both prism and gratings operate from mechanical cams of which the cam follower spacing pins can he adjusted for calibration purposes.

We have discovered that if the prism cam for the range 2000-8000 cm<sup>-1</sup> be adjusted, by the usual procedure for calibration adjustment, to scan the range  $1920-7100 \text{ cm}^{-1}$ , the first grating (which in the third order would have from its regular cam a nominal range 1950-6450 cm<sup>-1</sup>) is ganged with the adjusted prism very efficiently in the range 3000- $4500 \text{ cm}^{-1}$ . The available energy is such that spectral slit widths of less than 3 cm<sup>-1</sup> (see figure) are easily achieved with conveniently rapid scanning speeds. Frequency calibration for the evacuated prism-grating double monochromator is entirely unnecessary because the frequency in the third order is precisely three times the frequency for the first order read from the counter in the range  $650-2150 \text{ cm}^{-1}$ . with counter gear-box position 2. Fortuitously the grating slit programme 1 produces an acceptable energy level over the most useful portion of the extended range, 3300- $4200 \text{ cm}^{-1}$ . Stray light has been tested and found to be immeasurably small, as would be expected because the prism monochromator bandpass of 100-300 cm<sup>-1</sup> is small relative to the  $1250 \text{ cm}^{-1}$  interval between orders.

By offsetting the prism cam in the other direction, the fourth order of the grating could be brought into coincidence. The usable range of the double monochromator thus obtained would be limited to about  $4300-5700 \text{ cm}^{-1}$ , which is less useful for general analytical purposes.

The only disadvantage of the modification employing the third order is the loss of the region from  $7100-8000 \text{ cm}^{-1}$  of the NaCl prism range. Since this region is rarely consulted, particularly due to the bandwidths of 200-400 cm<sup>-1</sup>

obtained with the prism alone, the extension of the grating range as described above can serve only to increase the utility of the SP100 spectrophotometer.



Atmospheric water vapour A, 3744 • 5 cm<sup>-1</sup>; B, 3749 • 9 cm<sup>-1</sup>; C, 3752 • 2 cm<sup>-1</sup>.

The author would like to acknowledge the valuable assistance of Mr. T. C. Fletcher in effecting these modifications.

#### References

PIMENTEL, G. C., and McClellan, A. L., 1960, The Hydrogen Bond (San Francisco: Freeman), Chap. 3.

TARBET, C. S., and DALY, E. F., 1959, J. Opt. Soc. Amer., 46, 603.

J. SCI. INSTRUM., 1965, VOL 42

# A jacketed cell for the Bendix NPL polarimeter

### L. K. DALTON

Sugar Research Laboratory, Commonwealth Scientific and Industrial Research Organization, Biochemistry Department, University of Melbourne, Australia *MS. received 1st December 1964* 

Abstract. A cell of simplified construction, which is suitable for thermostatic control, has been designed for the Bendix NPL Automatic Polarimeter 143.

Because of its high sensitivity the Bendix NPL Automatic Polarimeter 143 (Bendix Electronics Ltd.) provides the means of measuring very small optical rotations (Gates 1958). When linked with a recording potentiometer it can

be used to follow reactions which involve a change in optical rotation, and even for single readings the recorder enables fuller use of the sensitivity of the instrument.

However, although the high sensitivity of the instrument