Pramāṇa, Vol. 5, No. 6, 1975, pp. 328–338. © Printed in India.

Normalisation and assessment of neutron diffraction data from liquids

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MS received 27 March 1975; in revised form 18 August 1975

Abstract. A couple of normalisation criteria have been proposed to assess liquid structure data over the past decade. In this paper, a critical examination of these and other plausible criteria is made. Neutron diffraction data from liquid krypton measured by Clayton and Heaton is used to study these criteria. It is shown that if the structure factor $S(Q) = 1 + \gamma(Q)$ satisfies the Krogh-Moe relation, namely

$\rho_0 = -\frac{1}{2\pi^2}\int_0^\infty Q^2 \gamma(Q)\, dQ$ where Q is the wavevector transfer and ρ_0 the mean atomic number density, $S(Q)$ is properly normalised. Subjecting such data to the other criteria would reveal, however, the quality of data in so far as systematic errors and/or termination errors are concerned. If the data is measured with reasonable accuracy to sufficiently large values of the wavevector transfer all these criteria would assess the quality of the data to almost the same extent. This is established in this paper by improving and extending the liquid krypton data by procedures similar to that of Kaplow, Strong and Averbach and subjecting this revised data to the various criteria. It is concluded, thereby, that (i) one can study the neutron diffraction data from liquids using any of the criteria mentioned in the paper to improve the quality of the data and (ii) for normalisation purposes the simple relation $\rho_0 = -\frac{1}{2\pi^2}\int_0^\infty Q^2 \gamma(Q)\, dQ$ would suffice.

Keywords. Neutron diffraction; liquids; structure factor.

1. Introduction

The determination of static and dynamic short-range correlation of atoms in a monatomic liquid forms an important aspect of the study of the liquid state. To get the pair correlation function one measures the neutron or x-ray diffraction pattern and obtains the normalised diffracted intensity $S(Q) = 1 + \gamma(Q)$, generally referred to as the structure factor, as a function of the wavevector transfer Q ($= 4\pi \sin\theta/\lambda$ in standard notation). The measured *coherently* scattered intensity $I(Q)$ is given by,

$$I(Q) = Nf^2(Q)\,[1 + \gamma(Q)]; \quad \text{x-rays} \tag{1 a}$$

and

$$I(Q) = I_\infty\,[1 + \gamma(Q)]; \quad \text{neutrons} \tag{1 b}$$

where $f(Q)$ denotes the atomic scattering factor and N and I_∞ are normalisation constants. The static pair correlation function $g(r)$ is obtained by a Fourier transformation of the structure factor:

$$g(r) = 1 + \frac{1}{2\pi^2 \rho_0} \int_0^\infty Q^2 \gamma(Q) \frac{\sin Qr}{Qr} dQ \tag{2}$$

where ρ_0 is the mean atomic number density of the liquid.

In practice, $S(Q)$ derived from an experiment is available only up to a maximum wave vector transfer, Q_{max}. Further it has statistical errors and may suffer from errors introduced during the processing of the measured intensity to obtain $S(Q)$. One of the systematic errors is the improper normalisation of the measured data. These limitations of $S(Q)$ lead to termination and other errors in $g(r)$. However, certain exact relations that $g(r)$ has to satisfy can be used to assess, correct and improve the structure factor. Basically all these relations arise due to the hard-core repulsion between the first neighbours in the liquid. Since, no two atoms can penetrate within a certain distance of closest approach, r_c, the probability of finding a second atom from a given atom is zero within this distance Mathematically,

$$g(r) = 0 \text{ for } r < r_c \tag{3}$$

Therefore, for distances $L < r_c$, eq. (2) reduces to,

$$\rho_0 = -\frac{1}{2\pi^2} \int_0^\infty Q^2 \gamma(Q) \frac{\sin QL}{QL} dQ \qquad \textit{Test I} \tag{4}$$

and for $L = 0$, we have the simple relation

$$\rho_0 = -\frac{1}{2\pi^2} \int_0^\infty Q^2 \gamma(Q)\, dQ. \tag{5}$$

This relation is well known [Krogh-Moe (1956) and Norman (1957)] and has been used extensively for normalising experimental data just by a knowledge of the mean atomic density.

A few years ago, Rahman (1965) derived an equation [*see* eq. (7) below] which structure factors should satisfy and suggested that this equation be used as a criterion particularly for normalisation purposes. Recently, Mountain (1972) has derived another relation [eq. (8) below] which could also be used for estimating the overall uncertainty in diffraction data.

Several workers [Randolph and Singwi (1966), Ocken and Wagner (1966), Greenfield, Wellendorf and Wiser (1971), de Graaf and Mozer (1971) and Wagner (1972)] have used the criterion suggested by Rahman (1965) to test diffraction data. Soon after the publication of Rahman's paper, we studied the implications of results of his paper in detail, particularly in relation to structure factor data of liquid krypton of Clayton and Heaton (1961) and concluded (Rao 1968) that the simple eq. (5) embodies in itself the best possible normalisation criterion and there was little additional improvement obtained by use of Rahman's criterion. A

mention of this conclusion has already been made by one of the present authors (Dasannacharya *et al* 1968). Since we continue to find several workers appealing to the criterion given in Rahman's paper, we think it worthwhile to report the details of our earlier investigations. In addition, we report now the results of applying Mountain's criterion to diffraction data of liquid krypton.

In section 2, we briefly outline various criteria (tests) found in literature and some new ones for studying liquid diffraction data. Some of these criteria are utilised in section 3 to examine Clayton and Heaton's neutron diffraction data on liquid krypton and the criteria are intercompared. In section 4, the structure factor listed by Clayton and Heaton is corrected and extended and in section 5, the tests are intercompared with the help of the extended data. A summary is given in section 6.

2. Criteria for diffraction data

Let us consider eq. (4) which is valid for any $L < r_c$. This itself gives the simplest test condition for assessment of data (test I) and is basically a general Krogh-Moe relation. Instead of demanding that $g(r)$ be zero, as in the above test, one could instead ask for $\int_0^L 4\pi r^2 g(r)\,dr$ to be zero. This leads to another test condition,

$$\rho_0 = -\frac{1}{2\pi^2}\int_0^\infty Q^2\gamma(Q)\left\{\frac{3(\sin QL - QL\cos QL)}{Q^3L^3}\right\}dQ \qquad \textit{Test II} \qquad (6)$$

Note that $L = 0$ in test II (as in test I) gives eq. (5), the Krogh-Moe normalisation relation. One could obtain a family of such relations* by using various functions for multiplying $g(r)$, integrating the resulting function over a distance $L < r_c$ and equating the integral to zero. Rahman (1965) has used a function $e^{-i\mu.r}$ and obtained the relation,

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* A few other simple test relations can be derived using the relation $\int_0^L r^n g(r)\,dr = 0$ where n is an integer., *e.g.*, $\int_0^L r\,g(r)\,dr = 0$ leads to the equation,

$$\rho_0 = -\frac{1}{2\pi^2}\int_0^\infty Q^2\,\gamma(Q)\left\{\frac{2(1-\cos QL)}{Q^2L^2}\right\}\,dQ \qquad (6\,a)$$

Similarly, $\int_0^L r^3 g(r)\,dr = 0$, leads to the equation,

$$\rho_0 = -\frac{1}{2\pi^2}\int_0^\infty Q^2\gamma(Q)\,\frac{4\{2(\cos QL - 1) + 2QL\sin QL - Q^2L^2\cos QL\}}{Q^4L^4}\,dQ \qquad (6b)$$

Since the multiplying function can be arbitrary, not all of them have a physical meaning.

$$\rho_0 j_1(\mu L) = -\frac{1}{2\pi^2}\int_0^\infty Q^2\gamma(Q)\left\{\frac{j_0[(Q-\mu)L] - j_0[(Q+\mu)L]}{2QL}\right\}dQ$$

$$\textit{Test III} \qquad (7)$$

where j_0 and j_1, are spherical Bessel functions and μ is an arbitrary parameter. Note that $\mu = 0$ in test III gives test II.

Mountain (1972) obtains his relation by requiring $\frac{d}{dr} g(r)$ to be zero for $L < r_c$:

$$\rho_0 = -\frac{1}{2\pi^2} \int_0^\infty Q^2 \gamma(Q) \cos QL \, dQ \qquad \textit{Test IV} \qquad (8)$$

Once again this equation is also a member of a family of relations obtainable by differentation as pointed out by Mountain. According to him eq. (8) is expected to be more sensitive to small defects in $\gamma(Q)$ than is the integration test, eq. (7).

It is obvious from the form of the test equations that they are all just different ways of weighting the function $Q^2 \gamma(Q)$ before integrating over Q. The weighting functions have a value of unity at $Q = 0$ and for larger values of Q all of them except the function in test IV decrease in value and asymptotically reach zero at infinite Q, oscillating in the intermediate region. The damping and the period of oscillation of the functions are decided by the choice of L. Sensitivity of a test depends on the rate of damping of the weighting function. Since the weighting function of test IV does not have a damped behaviour it may be expected to be the most sensitive of the ones listed here.

3. Test case of liquid krypton diffraction data

Rahman (1965) used both the x-ray (Gingrich and Tompson 1962) and neutron (Henshaw 1957) data on liquid argon and using test III concluded that the neutron data is much more reliable in the case of liquid argon since the x-ray data does not satisfy test III as well as the neutron data for two values of L.

We studied the liquid krypton data of Clayton and Heaton (1961) as a test case. Figure 1 shows the experimental neutron diffraction data $I(Q)$ from the liquid at 117° K and 0·8 atmosphere pressure and the data is tabulated in table 1 as a function of $Q/4\pi$. The data corresponds to an averaged curve through at least three complete diffraction patterns of the liquid under the same conditions of temperature and pressure and has been corrected for background, sample holder scattering and incoherent scattering but not for self absorption and multip scattering. The normalised intensity is obtained using eq. (1 *b*). Two asymptotic values of intensity I_∞ equal to 2·51 and 2·518 were considered for purposes of normalisation. $I_\infty = 2 \cdot 518$ satisfied the Krogh-Moe relation to a better degree than the value of 2·51, given by Clayton and Heaton.

We wish to assess the data in the light of test III, which depends on L and μ. Before we do this, we shall examine the data using test II. In figure 2, the right hand side of eq. (6) (test II) is plotted using $I_\infty = 2 \cdot 51$ and $2 \cdot 518$. The line corresponding to $\rho_0 = 0 \cdot 0176$ atoms/Å^3, the density of the liquid at 117° K and 0·8 atmosphere (Clayton and Heaton 1961), is also shown. This figure brings out certain interesting features:

(i) the sensitivity, defined as the change in the value of the integral for a small change in I_∞ is maximum at $L = 0$ and decreases with increasing L, so much so of that beyond $L = 1$ Å the test is completely insensitive to the two chosen values of I_∞.

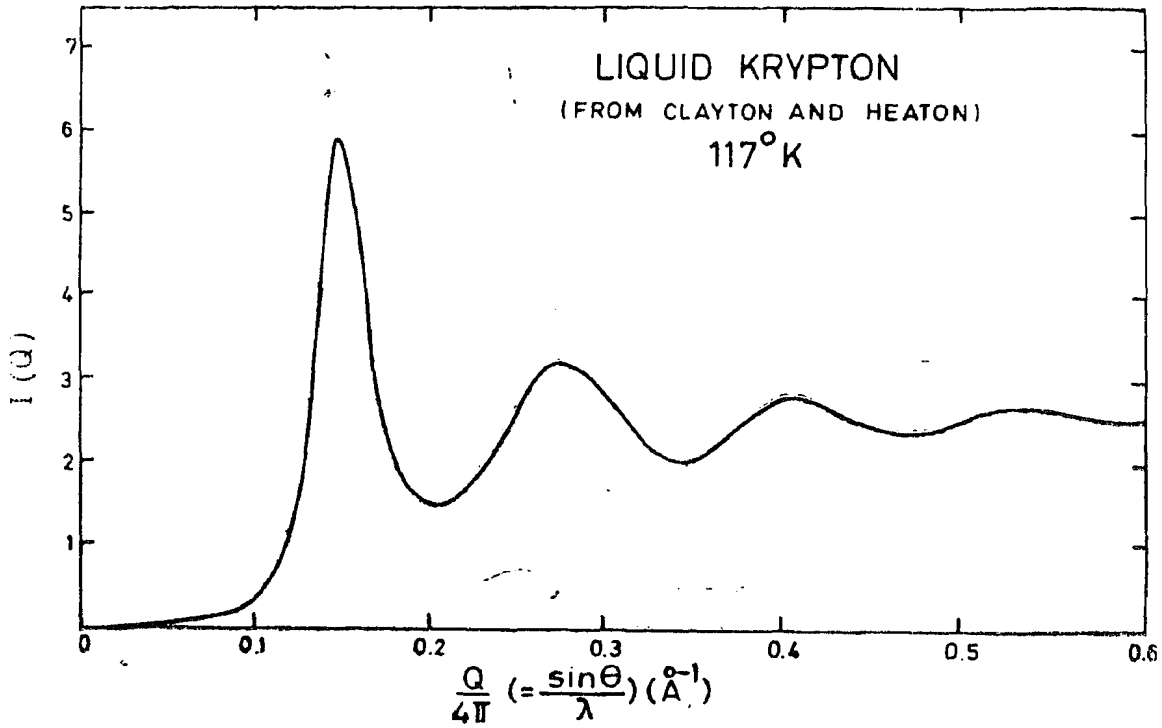

Figure 1. Coherently diffracted neutron intensity from liquid krypton at 117° K and 0·8 atmosphere as a function of wavevector transfer. [Clayton and Heaton (1961).]

Table 1. Neutron diffraction pattern from liquid krypton at 117° K [Based on ANL–6112 by Clayton and Heaton (1961)].

$Q/4\pi$	0	0·01	0·02	0·03	0·035	0·04	0·045	0·05	0·055	0·06	0·065	0·07	0·075	0·08	0·09
0	0·00	0·02	0·04	0·06		0·08		0·10		0·11		0·14		0·18	0·23
0·1	0·36	0·61	1·20	2·47	3·60	4·87	5·73	5·77	5·20	4·33	3·48	2·73	2·28	1·97	1·65
0·2	1·52	1·51	1·63	1·88		2·23		2·65		3·03		3·18		3·18	3·05
0·3	2·82	2·56	2·31	2·11		2·00		2·02		2·13		2·30		2·49	2·65
0·4	2·77	2·79	2·72	2·62		2·53		2·45		2·39		2·36		2·37	2·42
0·5	2·50	2·58	2·66	2·68		2·66		2·62		2·57		2·52		2·51	2·51

(ii) the value of the integral as a function of L is not constant as demanded by the test with any I_∞ reflecting the inadequacy due to termination at Q_{max} and/or inaccuracy of the data.

(iii) for certain L values the integral happens to be equal to the number density. We will show that for these L values and/or for L values very close to these (say, $L = 0{\cdot}1$, $0{\cdot}375$ and $2{\cdot}0$ marked by arrows in figure 2) test III will also be satisfied for all μ's at least as well as test II. At other L values (say at $L = 0{\cdot}7$), test III likewise will not be satisfied.

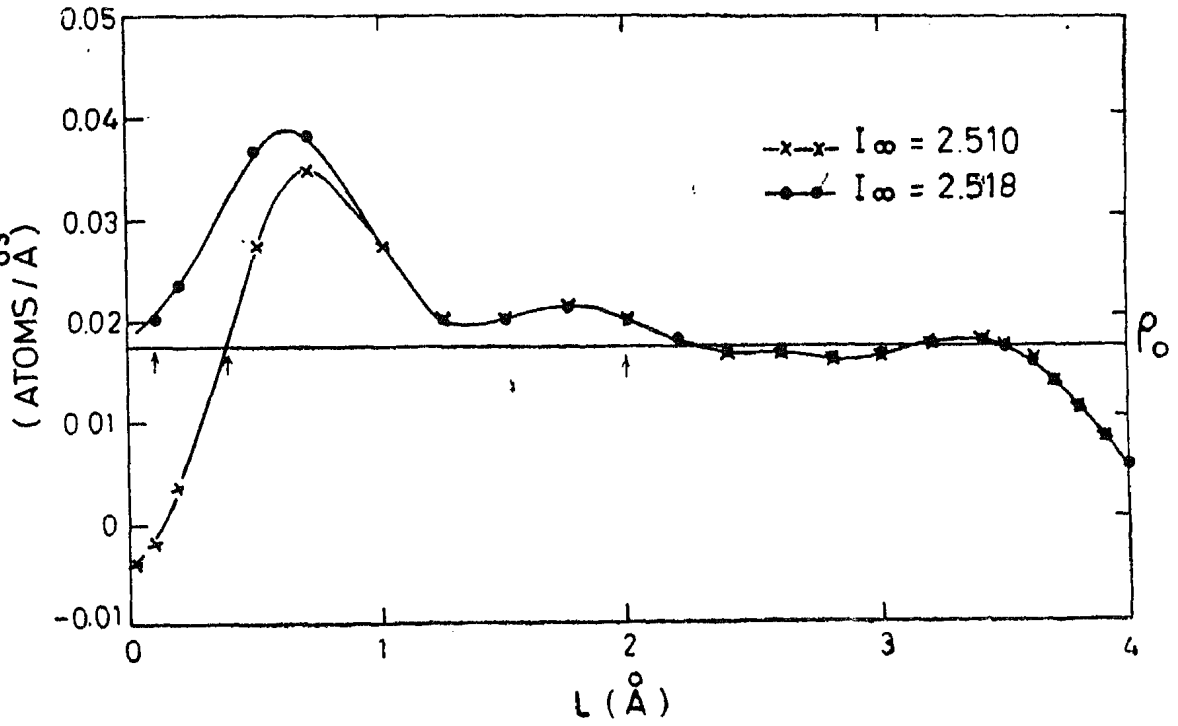

Figure 2. Test II applied to neutron diffraction data from liquid krypton shown in figure 1. The ordinate is value of the integral occurring on the right hand side in eq. (6) (*see* text) and the abscissa is the parameter L.

For this purpose, the left hand side $S_l(L, \mu)$ and right hand side $S_r(L, \mu)$ o eq. (7) (test III) have been plotted in figure 3 for $L = 0{\cdot}1$, $0{\cdot}375$, $0{\cdot}7$ and $2{\cdot}0$ as a function of μ for both $I_\infty = 2{\cdot}51$ and $2{\cdot}518$. We note that whenever test II, which, we remind ourselves, is just the special case, with $\mu = 0$ of test III, is satisfied the left and right hand sides are in agreement for the entire μ range (*e.g.*, at $L = 0{\cdot}1$ Å with $I_\infty = 2{\cdot}518$, at $L = 0{\cdot}375$ Å with $I_\infty = 2{\cdot}51$ and at $L = 2{\cdot}0$ Å with both the $I'_\infty s$). For $L = 0{\cdot}7$ Å both $I'_\infty s$ show disagreement. Thus we conclude that μ is an unnecessary parameter.

To conclude this section, test III reduces to test II as $\mu \to 0$ and in turn, test II reduces to the Krogh-Moe relation as $L \to 0$ giving the most sensitive of all normalisation conditions. However, an examination of the diffraction data subjected to a criterion given by test II, or any of the others, and a plot as shown in figure 2 does help to bring out the 'quality' of the data as will be demonstrated in the following section.

4. Refined liquid krypton data

Having found that the neutron diffraction data on liquid krypton as measured up to $Q_{max} \sim 7{\cdot}5$ Å^{-1} does not give constant value of the integral in eq. (6) as a function of L we decided to extend the data to larger Q values as done by Kaplow, Strong and Averbach (1965) for *Pb* and *Hg*. The aim of such an exercise is to arrive, from the experimental data, $S^E(Q)$, at a $g(r)$ which satisfies the following two physical conditions: (i) The Fourier transform of $g(r)$ must reproduce the normalised intensity up to Q_{max} within experimental errors and (ii) $g(r)$ must be zero below its first peak (for $r < r_c$)—that is, $g(r)$ must be free from ripples arising from truncation errors in Fourier transforming $S^E(Q)$. Extension of $S(Q)$ can be done, following Kaplow *et al* (1965), if $g^E(r)$ is corrected by removing "obvious irregularities and oscillations" in it to produce a new function $g^c(r)$.

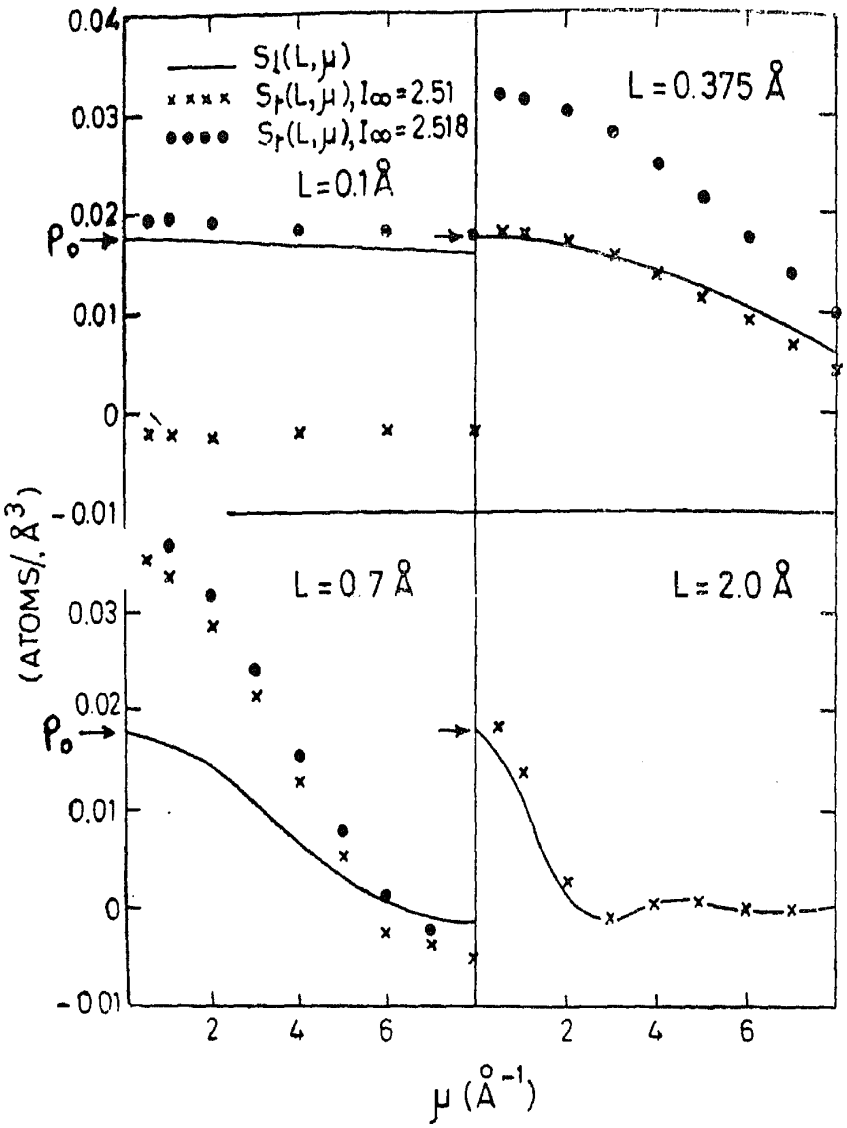

Figure 3. Test III applied to neutron diffraction data from liquid krypton shown in figure 1. The ordinates are the values of the left [S_I (L, μ)] and right [S_F (L, μ)] sides of eq. (7) plotted as a function of μ for various values of the parameter L for two I_∞'s, 2·51 and 2·518. Note that S_I (L, μ) is independent of choice of I_∞. At $L = 2{\cdot}0$ Å, only crosses are shown corresponding to $I_\infty = 2{\cdot}51$. Values for $I_\infty = 2{\cdot}518$ coincide with the crosses.

Using $g^e(r)$ one obtains a structure factor which extends beyond Q_{max}. This is used for the next cycle of Fourier transformations. Thus after a few iteration one should be able to arrive at a final corrected structure factor $S^e(Q)$ which satisfies the above mentioned criteria, if the original $S^E(Q)$ does not have significant systematic errors.

With the structure factor as listed by Clayton and Heaton it was not possible to achieve this self consistent set of $S^e(Q)$ and $g^e(r)$ which would fully reproduce the listed structure factor and ripple free $g(r)$. Hence we adopted two slightly different variations of the method of Kaplow *et al.*

In one we accepted a structure factor, $S_I^e(Q)$, which shows slight differences compared to the listed values of Clayton and Heaton. This is shown by the dashed lines in figure 4 where the full line below $Q = 7{\cdot}5$ Å^{-1} gives the listed $S(Q)$. The maximum difference between $S_I^e(Q)$ and $S^E(Q)$ amounts to $\sim 5\%$. This should be seen in relation to the fact that it is extremely difficult to get such

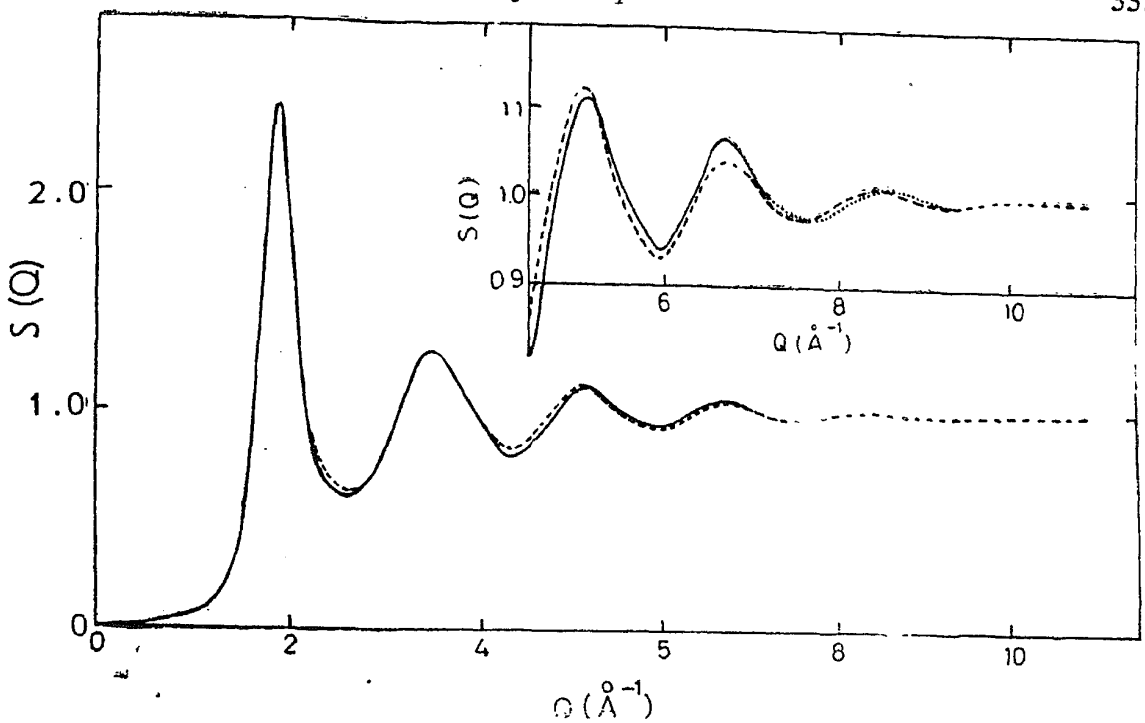

Figure 4. 'Corrected' and extended structure factor on liquid krypton using procedures outlined in the text. The full line indicates $S^E(Q)$, The experimental structure factor obtainable from listed data in Table 1 and shown in Fig. 1. Dashed line shows $S_I^e(Q)$; it departs slightly from $S^E(Q)$ over the measured range. Dotted line extends $S^E(Q)$ to yield $S_{II}^e(Q)$.

accuracies in neutron diffraction measurements*. We believe that this difference could be considered to be within experimental accuracies. If this view is accepted then the $S_e^I(Q)$ and $S^E(Q)$ (listed) may be considered to "match" with each other and the method is consistent with that of Kaplow *et al.***

In the second variation the listed $S^E(Q)$ is accepted without any change whatsoever but the data is extended as shown by the dotted line in figure 4 to obtain $S_{II}^e(Q)$. This, of course, matches $S^E(Q)$ below Q_{max} but does not produce a $g(r)$ which is as ripple free as that produced by $S_I^e(Q)$. We will demonstrate in the next section that $S_I^e(Q)$ is indeed the more acceptable structure factor of the two, whatever test criterion one may use to evaluate them.

5. Comparison of tests with data before and after refining

Structure factors $S_I^e(Q)$ and $S_{II}^e(Q)$ were both examined using test II and results are shown in figure 5. (Note that figure 2 and figure 5 are both given on the

* One may for example see the earlier measurements of Caglioti *et al* (1967), Ascarelli (1966) or North *et al* (1968) to get an idea of the typical error in $S(Q)$ measured by neutrons. Clayton and Heaton (1961) do not quote the magnitude of errors in their data. Another example may be found in a report by Page (1972) where the recently measured $S(Q)$ for liquid argon at 84·5° K are compared with those measured in 1968 by Yarnell. Although differences of order of 6% are found near the first and second maxima and the minima, the agreement is said to be reasonable. Even in these careful measurements, the overall agreement is only of the order of 3%. It is only recently that structure factors are obtained with an overall accuracy of ± 2%.

** A few other studies based on similar procedure have also indicated that differences are noticeable between $S^E(Q)$ and $S^e(Q)$ over the measured Q-range. See, for example, Ocken and Wagner (1966), Halder and Wagner (1966), Henniger, Buschert and Heaton (1967) and Cohen and Campbell (1972).

Table 2. Structure factor, S_I^c (Q) of liquid krypton extended by method of Kaplow, Strong and Averbach (1965).

Q	0·0	0·1	0·2	0·3	0·4	0·5	0·6	0·7	0·8	0·9
0.	0·0	0·01	0·01	0·02	0·02	0·03	0·03	0·04	0·05	0·06
1.	0·07	0·10	0·12	0·20	0·28	0·50	0·84	1·50	2·248	2·313
2.	1·848	1·308	0·960	0·796	0·698	0·652	0·631	0·643	0·694	0·762
3.	0·857	0·985	1·110	1·208	1·246	1·256	1·224	1·166	1·0943	1·0174
4.	0·9454	0·8860	0·8440	0·8280	0·8420	0·8750	0·9244	0·9785	1·0329	1·0809
5	1·1160	1·1260	1·1080	1·0738	1·0370	1·0045	0·9778	0·9560	0·9396	0·9311
6.	0·9342	0·9465	0·9645	0·9874	0·9884	1·0313	1·0407	1·0425	1·0392	1·0317
7.	1·0206	1·0079	0·9957	0·9868	0·9806	0·9772	0·9766	0·9802	0·9868	0·9938
8.	0·9992	1·0038	1·0077	1·0106	1·0121	1·0113	1·0096	1·0076	1·0047	1·0007
9.	0·9973	0·9948	0·9934	0·9934	0·9946	0·9938	0·9975	0·9988	1·0000	1·0010
10.	1·0018	1·0024	1·0028	1·0029	1·0026	1·0020	1·0012	1·0007	1·0003	1·0000
11.	1·0000									

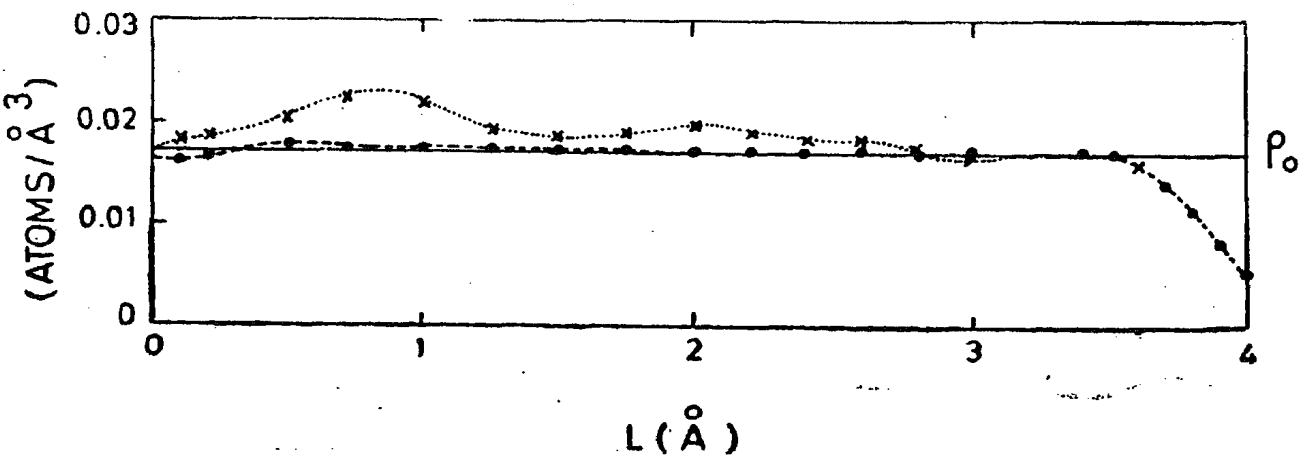

Figure 5. Test II applied to data given in figure 4. The dashed and the dotted lines correspond to $S_I^c(Q)$ and $S_{II}^c(Q)$ respectively.

same scale). It is easily observed from this figure that S_{II}^c does not satisfy test II to the same extent as $S_I^c(Q)$. We believe, therefore, that there are small errors in the listed $S(Q)$ which have now been corrected in $S_I^c(Q)$. $S_I^c(Q)$ is tabulated in table 2.

To compare the different tests, we have studied $S^E(Q)$, the raw data and $S_I^c(Q)$, the extended and corrected data using tests I, II and IV. The results of these calculations are shown in figure 6 where the integrals that occur on the right hand sides of eq. (4) (test I), eq. (6) (test II) and eq. (8) (test IV) are shown as functions of L. The line at 0·0176 of the ordinate gives the number density corresponding to the left hand side. Test III is not studied as it is already shown that μ is a redundant parameter in this test and test II replaces test III in the limit

$\mu \to 0$. Figure 6 (*a*) corresponds to $S^E(Q)$ and figure 6 (*b*) to $S_I^c(Q)$. It is obvious from figure 6 (*b*) that the oscillations, which one notices in figure 6 (*a*), get considerably reduced for *all* the tests when the data is refined. Beyond $L = 3{\cdot}4$ Å or so, all the tests depart considerably from their small oscillations indicating that we are approaching the critical radius r_c. The critical radius for krypton, obtained from law of corresponding states, is about 3·28 Å.

6. Summary

Normalisation and assessment of neutron diffraction data from liquids are important aspects of processing of such data. Several criteria are suggested in the literature for this purpose. In this paper we have examined the neutron diffraction data from liquid krypton using these criteria. As a result of these studies we find that Krogh-Moe relation (eq. 5) serves well for normalisation. Assessment of data with regard to systematic errors, termination and normalisation could be done by using any one of several tests mentioned in this paper. The simplest of course, is to just look for a ripple-free $g(r)$. Diffraction data can be refined

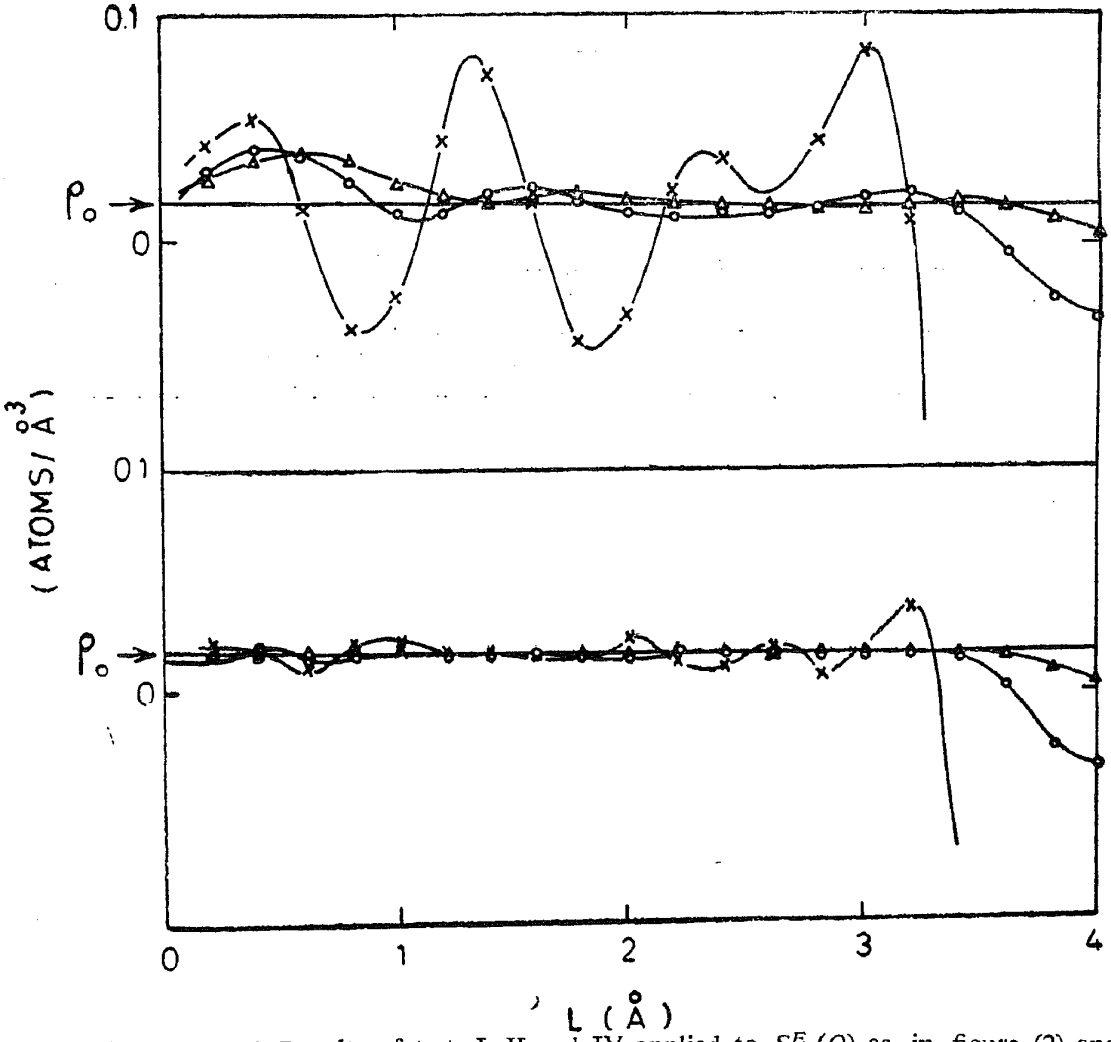

Figure 6. (*a*) Results of tests I, II and IV applied to $S^E(Q)$ as in figure (2) and indicated by ○, △ and × respectively.
(*b*) Results of tests I, II and IV applied to $S_I^e(Q)$ as in figure 6 (*a*) above.

by the method of Kaplow *et al* and when such a data is subjected to these tests one observes reduction in the amplitude of oscillations referred to earlier.

Acknowledgement

We are grateful to P K Iyengar for his interest in this work. The suggestions of the referees are also acknowledged.

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