Determination of respirable-sized crystalline silica in different ambient environments in the United Kingdom with a mobile high flow rate sampler utilising porous foams to achieve the required particle size selection

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A B S T R A C T

Inhalation of respirable crystalline silica (RCS) can cause diseases including silicosis and cancer. Levels of RCS close to an emission source are measured but little is known about the wider ambient exposure from industry emissions or natural sources. The aim of this work is to report the RCS concentrations obtained from a variety of ambient environments using a new mobile respirable (PM4) sampler. A mobile battery powered high flow rate (52 L min⁻¹) sampler was developed and evaluated for particulate aerosol sampling employing foams to select the respirable particle size fraction. Sampling was conducted in the United Kingdom at site boundaries surrounding seven urban construction and demolition and five sand quarry sites. These are compared with data from twelve urban aerosol samples and from repeat measurements from a base line study at a single rural site. The 50% particle size penetration (d50) through the foam was 4.3 μm. Over 85% of predict bias values were with ± 10% of the respirable convention, which is based on a log normal curve. Results for RCS from all construction and quarry activities are generally low with a 95th percentile of 11 μg m⁻³. Eighty percent of results were less than the health benchmark value of 3 μg m⁻³ used in some states in America for ambient concentrations. The power cutting of brick and the largest demolition activities gave the highest construction levels. Measured urban background RCS levels were typically below 0.3 μg m⁻³ and the median RCS level, at a rural background location, was 0.02 μg m⁻³. These reported ambient RCS concentrations may provide useful baseline values to assess the wider impact of fugitive, RCS containing, dust emissions into the wider environment.

1. Introduction

Crystalline silica is an abundant mineral found in many clays, rocks and sands and is widely used in building materials, ceramics, chemicals, glass and metallurgical industries (Moore, 1999). In the workplace, the health related particle size range of interest for airborne crystalline silica measurements is termed the ‘respirable’ fraction i.e. particles that can penetrate deep into the gas-exchange regions of the lung. Respirable refers to a size range containing particles mostly less than 16 μm and is derived from a cumulative log normal distribution with a 50% penetration cut-off aerodynamic diameter (d50) of 4.00 μm (EN 481, 1993). The respirable dust fraction has a similar target d50 value to an environmental particle size fraction referred to as PM4. PM4 has a d50 penetration value that lies between particle size fractions of PM2.5 (d50 at 2.5 μm) and PM10 (d50 at 10 μm) more commonly employed in ambient air monitoring programs.

Exposure by inhalation to respirable crystalline silica (RCS) particles is a hazard encountered by those working with materials in construction and quarry industries and can result in a range of adverse health effects including silicosis (NIOSH, 2002) and lung cancers (IARC, 2012). In a retrospective study it was estimated that around 900 cases of occupational cancers in Great Britain in 2004 were attributable to exposure to RCS (Rushton et al., 2012). Crystalline silica is a common constituent of many natural and building materials and emissions of dust may also result in background exposures. In the United States (US) of America, an annual running population health benchmark exposure value of 3 μg m⁻³ is used in some states, for example, by California’s Office of Environmental Health Hazard Assessment (2005) and in Minnesota, by their Department of Health (MDH, 2017) for the monitoring of sand extraction activities (MDH, 2017). This benchmark value is based on an evaluation conducted by the US Environmental Protection Agency (EPA, 1996) and is an average value estimated from PM10 sampling data at which there is thought to be little or no risk to the wider populous. RCS concentrations of 10 μg m⁻³ are also significant because they are potentially detectable (for an 8 h sample) with the personal respirable sampling.

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The aim of this article is two-fold. Firstly, to present information on the development of an aerosol sampler that employs foams of known porosity to achieve the desired respirable (PM<sub>10</sub>) particle size cut. Foams provide a low cost and light weight modification as an insert into the inlet of an existing sampler (Kenny et al., 2001a). They provide a mechanism to select a smaller particle size than is captured by the inlet design and are frequently used in workplace personal exposure sampling (Chen et al. 1998; Aitken et al., 1993 and Kenny and Stancliffe, 1997). A useful attribute of foam systems is that a fraction of particles larger than the respirable fraction (inhalable dust) can also be measured i.e. by summing particles collected on the foam and particles that are collected on the filter. They have also been used for environmental exposure assessment. Mark et al. (1997) modified a personal workplace sampler with a foam insert to collect PM<sub>10</sub> to assess exposure of the public to atmospheric particulate. Foams were used in a CIS sampler, which has a conical inlet, for the high flow rate (16.5 L min<sup>−1</sup>) personal sampling of PM<sub>2.5</sub> (Adams et al., 2001).

The second aim is to present for the first time, measurements of RCS in urban air taken at the fence-line surrounding construction and demolition activities in Great Britain (GB) and to compare them with ambient measurements taken in quarries and at a rural site. Whilst there is abundant information regarding levels of worker exposure to RCS in the immediate vicinity of the emission (Chisholm, 1999; Lumens and Spee, 2001; Ehrlich et al., 2013; Esswein et al., 2013) in contrast, there is a paucity of information regarding measured concentration of RCS in the wider urban air environment. Fugitive dust emissions from construction activities are generally poorly quantified in global and national emissions estimates (Font et al., 2014), and lack comprehensive information on their chemical makeup and the RCS content is generally non-existent. There are a number of reasons why this is so: limit values for RCS in ambient air do not exist in many countries; measurements require specialised analytical facilities equipped with X-ray diffraction (XRD) instrumentation and there are no consensus on what particle size fraction (i.e. PM<sub>10/4/2.5</sub>) should be collected, unlike the workplace environment where respirable sampling is an established approach.

Some ambient aerosol studies have been done. Davies et al. (1984) measured crystalline silica in the in cities in California and found up to 1.9 μg m<sup>−3</sup> in the fine (PM<sub>2.5</sub>) and between 1 and 8 μg m<sup>−3</sup> in the course (PM<sub>10</sub>). In Rome, average weekly concentration values of crystalline silica in the PM<sub>10</sub> size fraction were reported as between 0.6 and 1.5 μg m<sup>−3</sup> (Puledda et al., 2003) and between 0.3 and 2.9 μg m<sup>−3</sup> (De Berardis et al., 2007). Increased silica concentrations were found to coincide with southerly winds suggesting that the periodic influx of sand particles transported from the Sahara was a contributing factor. Monitoring studies just beyond the fence line, at industrial locations have also been reported. Richards et al. (2009) measured ambient RCS (as PM<sub>4</sub>) around three Californian sand and gravel plants and reported downwind values in the range 0.3–2.8 μg m<sup>−3</sup>. Richards and Brozell (2015) achieved slightly lower RCS values for PM<sub>4</sub> when sampling for 24 h, around five sites producing sand for the fracking industry in Wisconsin (Range of geometric means 0.22–0.36 μg m<sup>−3</sup>, maximum 1.1 μg m<sup>−3</sup>) Peters et al. (2017) conducted respirable sampling at homes within 800 m of quarries extracting sand for fracking operations. Levels of exposure near seventeen homes were generally, less than 0.4 μg m<sup>−3</sup>. Some higher RCS concentrations were found (15–37 μg m<sup>−3</sup>) from some long term samples (less than three percent) when wind velocities were also elevated. Higher crystalline levels from PM<sub>10</sub> samplers (4–19 μg m<sup>−3</sup>) were found when monitoring mine tailings depots in South Africa containing 70–90% crystalline silica (Andraos et al., 2018). The mine tailing sites had a substantial proportion of ultrafine particles and the rainfall was limited (less than 130 mm each month).

Fig. 1. The HSE high flow rate mobile environmental respirable dust sampler in position at a quarry.

2. Experimental

2.1. Aerosol sampler development and validation

The aerosol sampler used in the work, shown in Figs. 1 and 2, was designed and constructed at the Health and Safety Executive’s laboratory. Two heavy duty leisure lead acid batteries were used to power a rotary vane pump (Rotheroe and Mitchell L60 model) allowing a nominal flow rate of 50 L min<sup>−1</sup> for a period of up to 12 h. Extended sampling operations using mains power supply were also possible. To achieve the desired respirable particle selectivity, a foam separator system was developed which consisted of a sandwich of a 20 mm thick reticulated polyurethane foam disk (45 pores per inch porosity) and a 10 mm thick foam disk (60 pores per inch porosity). The specifications for this foam separator were derived from the use of an empirical model developed by Kenny et al. (2001b) and subsequently validated by challenging representative foam separators with a test aerosol of glass microspheres of known median diameter in accordance with procedures set out in EN 13205-2 (CEN, 2014). The d<sub>50</sub> and the fractional penetration for the whole particle size distribution penetrating the foam particle selector were calculated and modelled. Further details are provided in Supplementary Information S1. The differences between the fitted performance curve and the target respirable convention were used to calculate sampler bias for an array of challenge size distributions, described in EN 13205:2 (CEN, 2014). A bias map was developed for each theoretical challenge dust to assess how closely the sampler would agree with the sampling convention for a range of exposure scenarios (Görner et al., 2001). The respirable dust fraction that penetrates the foam was subsequently collected onto a 2 μm pore size 60 mm diameter mixed cellulose ester (MCE) filter, which was selected as it ashes easily thus facilitating the recovery of RCS during subsequent analysis.

2.2. Sampling at quarry sites

Five quarries in England and Wales that handle quartz sand or sandstone were surveyed. The sites were selected because they employed good dust suppression techniques (e.g. wetting of lorry tyres).
Extracting sand was the dominant activity in three quarries, whilst extracting grit or sandstone and their subsequent crushing and screening was the dominant activity in the other two. Four samplers were placed in positions down wind of the site. A fifth sampler was placed upwind of the emissions and close to the site boundary to assess contributions from off-site RCS emissions. Data was collected for five sequential days at each site. Care was taken to ensure that the samplers were not too close to buildings and large obstructions (were > 3 m from nearby buildings) where dust levels could be affected by the re-circulating wake in the lee of the building or obstruction.

2.3. Sampling strategy at urban construction/demolition sites

Samplers were positioned at site boundaries in seven urban locations in the north of England, cognisant of the need not to impede worker activities. Construction activities investigated included demolition by mechanical means, general activities (including truck movement and digging), road building and power cutting of silica-containing bricks. The majority of sites were visited on two separate days (except brick cutting). At each site, one sampler was deployed at a location deemed to be upwind of the prevailing winds whilst two or three samplers were deployed downwind. A further sampler was placed in a mobile laboratory and deployed as a further upwind sampler to assess if the chosen upwind site boundary sampler was truly representative of background ambient levels. Where possible, for comparative purposes, this sampler was located next to a local authority or United Kingdom (UK) national monitoring station that deployed a Tapered Element Oscillating Microbalance (TEOM) real time PM10 sampler. Aerosol sampling over a working day (6–10 h) resulted in nominal sample volume of 18–30 m$^3$. Further sampling details can be found in the survey report (Stacey et al. 2011).

2.4. Sampling strategy at a rural background site

A single sampler was placed on a hillside 390 m above sea level on the Health and Safety Executive’s Laboratory site (Grid reference SK 05894 70241), which is on the edge of the town of Buxton in the Peak District in GB. Whilst quarrying is a nearby industrial activity this is mostly associated with limestone extraction, which has minimal silica content. Sampling took place at roughly weekly intervals, from January 2011 to March 2012, using a mains powered version of the sampler described in section 2.1. Sampling was not carried out every week due to inclement weather, equipment availability and staffing resources. In total, thirty four weekly filter samples were collected covering 46% of the available time interval and the average sampling interval was 6.6 days (475 m$^3$ air volume). Further opportunistic sampling was also conducted to capture possible deposition from the Grimsvötn volcano eruption (24th – 29th May 2012, 288 m$^3$ air volume sampled) and from a Saharan dust event that affected the UK (2nd - 5th April 2014, 214 m$^3$ air volume sampled). Further details are provided in Supplementary Information S3.

2.5. Laboratory analysis

Filter weighing was conducted in a balance room with controlled humidity and temperature using a balance with a readability of ± 1 μg (UX 6 Ultrabalance, Mettler-Toledo Ltd, Leicester, UK) to determine collected sample mass. Filter samples were then subjected to a low temperature plasma ashing (Emitec K1050X, Quorum Technologies Ltd, Ashford, UK), resuspension of RCS onto polycarbonate filters and subsequent analysis by XRD (Xpert pro MDP, Pan Analytical UK Ltd, Cambridge, UK). Procedures described in method 7500 (NIOSH, 2003) were followed. Quality control filters, prepared by spiking MCE filters with known quantities of a quartz reference material A9950 (HSE, Buxton, UK), were carried through the analytical procedure which gave mean recoveries of RCS of 92%. The analytical limit of detection was determined to be 6 μg RCS/filter equating to a method detection limit of 0.2 μg m$^{-3}$ for a nominal 30 m$^3$ air sample i.e. 10 h sampling at a nominal 52 L min$^{-1}$.
3. Results

3.1. Performance of the aerosol sampler

The testing of the foam combination to the respirable convention is detailed in Supplementary Information (S1). The mass median aerodynamic particle size diameter, at which the 50% particle size penetration diameter ($D_{50}$) was obtained for this foam sampler was measured as, 4.35 $\mu$m, which is within 9% of the specified 50% penetration cut-off value in EN 481 (CEN, 1993). It is common practice to assess the bias of an sampler for the respirable convention against a series of theoretical particle size distributions and geometric standard deviations following the procedure in EN13205:2 (CEN, 2014). More than 85% of the predicted sampling bias values were less than 10% over the respirable size range. An example of a bias map for the foam selector is shown in Fig. S3 (Supplementary Information S1). The operational reliability of the samplers during the site visits was good, provided the batteries were regularity charged and well maintained. However, sampler failures, due to ingress of water into the sampler head during periods of very inclement weather, were noted at the exposed rural sampling site.

3.2. RCS concentrations obtained from site visits

A list of sampling sites, numbers of samples collected and median RCS concentration data is presented in Table 1. The range of results from construction activities are compared with those obtained from a study involving five quarries (Supplementary Information S2). The number of results for each sampling site above the values of 10 and 3 $\mu$g m$^{-3}$ are also given in Table 1. To facilitate the calculation of summary statistics such as the median, a value of 0.5 x LOD was entered for all analytical results reported less than the LOD rather than to use zero. RCS concentrations obtained from weekly sampling at the rural site are provided in Supplementary Information S3.

Overall, the 95th percentile of RCS concentrations was 11 $\mu$g m$^{-3}$, which indicates that very few values are potentially detectable with less sensitive personal sampling equipment used in occupational hygiene. For example, 94% of results from the construction and quarry sites are below the value of 10 $\mu$g m$^{-3}$ and 79% are below the benchmark US value of 3 $\mu$g m$^{-3}$. These results are compared to those obtained at quarries. Similarly, 6% of RCS site boundary concentrations from sand production quarries (6 from 116) were greater than 10 $\mu$g m$^{-3}$ and 21% (25 from 116) were greater than 3 $\mu$g m$^{-3}$. The same data is presented graphically in Fig. 3 as box – whisker plots. The % of RCS in the collected respirable mass fraction (mass/mass) was also calculated for the various sites surveyed and is presented as a maximum-minimum median plot in Fig. 4.

The XRD technique was also used to interrogate other mineral phases present in the filter samples. The most common crystalline materials observed in ambient dust from the urban samples were, halite (NaCl), calcite (CaCO$_3$) and quartz (SiO$_2$). XRD reflections indicated the presence of clays (kaolinite (Al$_4$(Si$_8$O$_{20}$)(OH)$_{12}$) or illite) and maghemite (Fe$_2$O$_3$) in some samples. Significant amounts of material associated with concrete products were found in samples from the demolition sites i.e. anhydrite (CaSO$_4$) and calcium sulphate hydrate (CaSO$_4$·0.5H$_2$O) from dehydration of gypsum, dolomite (CaMg(CO$_3$)$_2$), portlandite (Ca(OH)$_2$), and halite (Ca(SiO$_3$)O). In contrast, few rural ambient samples showed the possible presence of salt (NaCl) and many contained sodium sulphate (Na$_2$SO$_4$) or sodium hydrogen sulphate (Na$_3$H(SO$_4$)$_2$).

| Classification     | Process                                      | Number of sampling sites | Number of samples taken (measured) | Upwind median (range) $\mu$g m$^{-3}$ | Downwind median (range) $\mu$g m$^{-3}$ | Number of RCS concentrations greater than 10 $\mu$g m$^{-3}$ | Number of RCS concentrations greater than 3 $\mu$g m$^{-3}$ |
|--------------------|----------------------------------------------|--------------------------|------------------------------------|----------------------------------------|----------------------------------------|------------------------------------------------|------------------------------------------------|
| Quarry             | Sandstone crushing, grading, bagging         | 1                        | 25 (24)                            | 0.65 (0.5–2.2)                         | 3.2 (0.2–21)                           | 4                                               | 9                                               |
| Quarry             | Sand and Gristone stone block production     | 1                        | 25 (20)                            | LOD (LOD – 0.6)                       | 1.15 (LOD – 20)                        | 2                                               | 6                                               |
| Quarry             | Sand Extraction, grading and bagging        | 2                        | 50 (48)                            | 2.5 (1.1–5.7)                         | 0.5 (LOD – 9.8)                        | 0                                               | 8                                               |
| Quarry             | Ripped sandstone to produce sand, grading, bagging | 1                        | 25 (22)                            | LOD (LOD – 2.9)                      | LOD (LOD – 2.9)                        | 0                                               | 0                                               |
| Construction       | Demolition                                  | 3                        | 22                                 | 0.5 (LOD – 7.4)                       | 1.24 (LOD – 11.5)                     | 2                                               | 7                                               |
| Construction       | Brick cutting with grinder                  | 2                        | 6                                  | 0.5–2.9                              | 6.15 (1.2–11.9)                        | 2                                               | 2                                               |
| Construction       | Road building                                | 1                        | 8                                  | LOD                                  | 0.69 (0.11–1.04)                       | 0                                               | 0                                               |
| Construction       | General Activities                          | 1                        | 8                                  | LOD                                  | 0.22 (0.13–0.39)                       | 0                                               | 0                                               |
| Urban air          | Ripped sandstone to produce sand, grading, bagging | 13                       | 12 (11)                            | (LOD – 0.3) – 60% of results at the LOD of 0.1 | 0.019 (LOD – 0.12) – 30% of results at the LOD 0.01 | 0                                               | 0                                               |
| Volcanic Ash       | Grimsvötn eruption event                    | 1                        | 1                                  | 0.033 (air volume 288 m$^3$)          | 54.4 (air volume 214 m$^3$)            | 1                                               | 1                                               |
| Sahara Dust        |                                              | 1                        | 1                                  | 0.34 (air volume 214 m$^3$)           | 54.4 (air volume 214 m$^3$)            | 1                                               | 1                                               |

4. Discussion

A mobile respirable sampler was successfully developed which enabled RCS samples to be collected in ambient settings at a flow rate of 52.1 min$^{-1}$. Laboratory validation work demonstrated that the foam separators, when challenged with test aerosols, were effective at segregating the requisite particle sizes. This enabled a method detection limit (MDL) of 0.3 $\mu$g m$^{-3}$ to be achieved for an 8-h sampling period i.e. covering a typical working shift, which was 10% of the US health benchmark value of 3 $\mu$g m$^{-3}$.

Results for RCS from all construction and quarry activities at site boundaries were generally low with a 95th percentile of 11 $\mu$g m$^{-3}$. Out of the 158 filter samples analysed, only 32 (about 20% of total) were found to exceed the annual health benchmark value of 3 $\mu$g m$^{-3}$ applied in some states in America for population exposure assessment purposes. Of these 32 samples, only 10 samples were found to exceed 10 $\mu$g m$^{-3}$ (6% of total), a value which is detectable with the sampling equipment commonly used when sampling workers for occupational hygiene assessments. These higher RCS concentrations were associated with activities that involved materials of high silica content coupled with energetic processes such as grinding and grading of stone in quarries or power cutting of brick during block paving. Overall, the data suggests that the dust suppression techniques (e.g. wetting of...
suppression was challenging because the building was taller than what operation it was observed that the application of a water spray for dust surfaces) can be effective and/or that dust dispersion, dilution occurs fairly rapidly away from the source of emissions.

That said there can be no room for complacency. At one demolition work, summarised in Fig. 4, suggests that a 10% value indeed ap- proximate to a median value in deriving estimated crystalline silica. This percentage was derived from earlier published background, from the Grimsvötn volcano eruption was noted at this developed parts of the UK in early April 2014 but no impact, above background, from the Grimsvötn volcano eruption was noted at this particular rural sampling site. This latter result is supported by Horwell et al. (2013) who determined that the crystalline silica content of this volcanic ash was minimal.

This was a limited study so it could be informative to undertake future RCS surveys, employing the methodology used here. For example, in the following situations: localized paving activities within a street canyon; regional dust deposition events such as sand blown in from the Sahara and monitoring around potential gas fracking sites where, in the US for example, high worker RCS exposures have been noted as a result of handling of sands used in the fracking process (Esswein et al., 2013). The XRD technique employed was used to interrogate sampled particles for other mineral phases present (Esteve et al., 2013). Examination of crystalline phases within samples could be useful in identifying the origins of dust emissions. For example, significant amounts of material associated with concrete in products were found in filter samples from the surveyed demolition sites. The rural filter samples were found to contain crystalline sodium sulphate (Na₂SO₄) or sodium hydrogen sulphate (Na₃H(SO₄)₂), indicative of the formation of secondary aerosols.

Further surveys could be informative but may be limited by available resources. Use of existing PM₁₀/₂.₅ monitoring data to estimate crystalline silica concentrations has been used by the US EPA (1996) who estimated a national annual urban air average of 3 μg m⁻³ crystalline silica. Here they used existing PM₁₀ mass monitoring data with the underlying assumption that, uppermost, 10% of this mass was crystalline silica. This percentage was derived from earlier published dust compositional measurements (Gift and Goldsmith. 1997). Our work, summarised in Fig. 4, suggests that a 10% value indeed approximates to a median value in deriving estimated crystalline silica concentrations around localized emission sources such as some sandstone quarry activities or brick cutting i.e. where high crystalline...
containing materials and energetic processes such as grinding or cutting are involved. A lower value of between 2.5 and 5% would perhaps be more appropriate for RCS concentration estimators around urban construction and demolition activities with a reduced value perhaps of about 1% for estimating background urban aerosol concentrations.

Making use of sampler assets already deployed in existing air monitoring networks is another possibility. Jones et al. (2001) have shown that it is possible to analyse filter samples from the TEOM (tapered element oscillating microbalance) PM<sub>10</sub>/2.5 samplers which are widely deployed in the UK air monitoring network. Here crystalline silica measurements could potentially be undertaken using either a direct-on-filter XRD approach as advocated in ISO 16258-1 (ISO, 2015) or by using a more sensitive direct-on-filter Raman method currently under development at HSE (Stacey et al., 2017).

5. Conclusion

This work set out to develop a mobile high flow rate sampler, with foam particle size selectors, which were used to undertake ambient RCS surveys where no mains electricity was available. The reported RCS concentrations obtained from these surveys were low. Only 5% of RCS concentrations were potentially detectable with the less sensitive sampling equipment and sampling flow rates routinely applied in occupational hygiene. The majority of these data (80%) were below the health guidance value of 3 μg m<sup>-3</sup> used in some states in the US, suggesting the impact of these emissions beyond the workplace is minimal. Emissions from work activities involving energetic processes e.g. rock crushing, milling, power saw cutting and largest scale demolitions, provided the largest proportions of crystalline silica in respirable dust that may, on occasion, approach or exceed a benchmark 10% upper threshold value established by the EPA in the US to predict crystalline silica in respirable dust. Most proportions of RCS in PM<sub>4</sub> were well below the 10% value for most of the sampling scenarios measured. The median rural background RCS level was 0.02 μg m<sup>-3</sup>, which indicates that the levels of RCS at the site in Buxton are low enough to be sensitive for the monitoring of large scale natural emission events. Given the transient nature of many construction activities, and provided that good dust suppression protocols are followed, then it is unlikely that levels of RCS in airborne dust emitted from construction sites would be sufficiently high and sustained so as to be of concern to the wider public.

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Appendix A. Supplementary data

Supplementary data related to this article can be found at http://dx.doi.org/10.1016/j.atmosenv.2018.03.032.

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Fig. 4. Plots of the highest, the median and the lowest percentages of respirable crystalline silica in respirable dust (% m/m) showing the range of the proportion of RCS recorded from worksite boundaries from natural sources and at a single rural location. The line of the chart shows the 10% EPA upper boundary level discussed by Gift and Goldsmith (1997).
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