PAPER

On-filter determination of collected wood dust by diffuse reflectance infrared Fourier-transform spectroscopy (DRIFTS)

Roy J. Rando,[a] Rachele A. Gibson,†[a] Cheol-Woong Kwon,[a] Halet G. Poovey‡[a] and Henry W. Glindmeyer[b]

[a] *Tulane University, School of Public Health and Tropical Medicine, Department of Environmental Health Science, New Orleans, LA, USA*
[b] *Tulane University, School of Medicine, Section of Pulmonary, Critical Care and Environmental Medicine, New Orleans, LA, USA*

Received 8th February 2005, Accepted 4th May 2005
First published as an Advance Article on the web 23rd May 2005

A new analytical technique based on DRIFTS spectroscopy has been developed for the specific and sensitive determination of size-fractionated wood dust from 37 mm glass fiber filter samples collected with the Respicon™ sampler. A translational diffuse reflectance apparatus was modified to accept filter samples by incorporating a special filter holder in the sample stage and a clockwork motor to drive the translational stage during infrared scanning, thus providing an average analysis across the filter face. Filter samples were pre-treated with ethyl acetate to uniformly redeposit dust onto the filter and extract potential chemical interferences. Two absorbance maxima (1251 and 1291 cm^{-1}), corresponding to the cellulose content of the wood, were suitable for quantitation of wood dust. Analysis of seven species of wood at 1291 cm^{-1} showed an equivalent quantitative response for all species except maple. The response at 1251 cm^{-1} was more variable across species but more sensitive for the softwoods. There was a statistically significant effect of particle size on the analytical response, so that analytical standards should be matched to the samples in terms of particle size distribution. Analytical limit of detection was approximately 0.08 mg of wood dust per sample with overall precision of about 6%. Comparison of DRIFTS and gravimetric analyses of 51 pure wood dust samples ranging from about 0.2 to 2 mg yielded a slope of 1.08 and r^2 equal to 0.9. Other particulate contaminants common in the industrial wood processing industry showed little or no interference with the determination of wood dust by this method.

Introduction

There is increasing interest in industrial wood dust exposure, particularly in light of putative effects on respiratory health and the risk of nasal cancer associated with chronic exposure.[1,2] Modern exposure assessment methods for wood dust generally involve filter collection of wood particles from the breathing zone of exposed workers utilizing various size-selective pre-samplers and sample inlets. These techniques, as well as less sophisticated techniques that collect so-called "total" particles, typically employ simple gravimetric analysis of collected particulate matter. Gravimetric analysis of collected particulate matter is non-specific and all particles captured on the filter, regardless of source, are included in the result. While in many industrial settings wood dust may be the most prominent particulate contaminant, other airborne substances such as road dust, tire rubber, tobacco and other smokes, and spray-finish aerosols may be present in significant quantities, resulting in an over-estimate of exposure to wood dust. In consideration of possible changes in the classification of wood dust from a nuisance dust to one that is specifically regulated, improved techniques to specifically identify and quantitate wood dust are needed.

Wood dust is a heterogeneous material composed of lignin, cellulose, hemicellulose, and various wood extractives including alkaloids, terpenes, fatty acids, *etc*. Some of these components, such as tannins and terpenes, have been evaluated for use as analytical markers in an attempt to quantify wood dust in the presence of other dusts. Tannins are found in all types of wood, but vary greatly in quantity between hard and softwood. This difference was used recently to estimate the content of oak in mixed wood dusts *via* determination of gallic acid in dust extracts.[3] Various mono- and di-terpenes and resin acids have also been examined as possible markers of exposure to wood dust, but their concentrations have been found to vary widely among species as well.[4,5] In contrast to these materials, which are relatively minor constituents of wood and thus subject to wide variation in content, cellulose, hemicellulose, and lignin comprise the majority of the bulk of wood dust. Softwoods contain on average 42% cellulose, 27% hemicellulose, and 28% lignin, whereas for hardwoods, the averages are 45%, 30%, and 20%, respectively.[6]

In this work, we have developed and evaluated a new method for quantifying wood dust using diffuse reflectance infrared Fourier-transform spectroscopy (DRIFTS). As shown in Fig. 1, an intense and prominent infrared absorption band in the region of 1200–1300 cm^{-1} is present in the DRIFTS spectra of wood dust due to its cellulose content. The method was developed by focusing on this absorption band as the basis of the analysis. The analytical and sample preparation techniques were developed specifically with reference to Respicon™ filter samples that are being collected in an on-going study of the lower respiratory health effects of dust exposure in the wood processing industries.[7] Thus, the ultimate aim of this work was the development of a convenient, sensitive, and specific method for direct, on-filter determination of the wood content of size-fractionated dusts collected from industrial environments.

Experimental

All work was performed on a Galaxy 5000 series Fourier-transform infrared spectrophotometer (Mattson Instruments, Inc., Madison, WI). The spectrometer was fitted with an

† Present address: Louisiana State University, School of Medicine, New Orleans, LA, USA.
‡ Present address: Nelson Architectural Engineers, Inc., Dallas, TX, USA.

DOI: 10.1039/b502025e

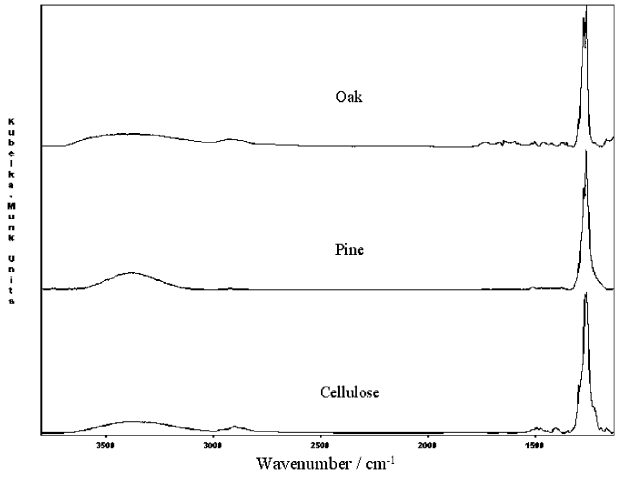

Fig. 1 DRIFTS spectra of reference materials.

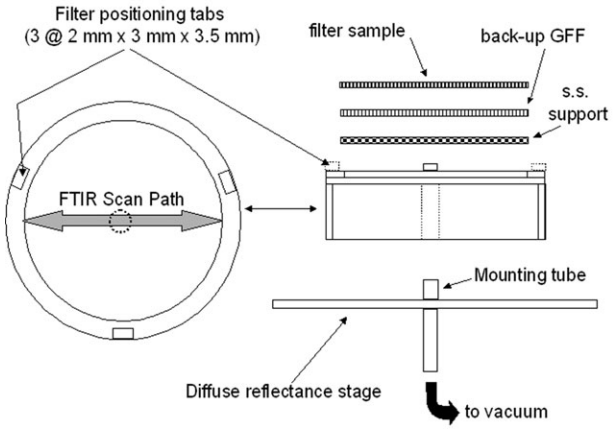

Fig. 2 Schematic of modified stage and filter holder for DRIFTS analysis of Respicon™ filter samples.

external sampling compartment that housed the sampling apparatus and a liquid nitrogen-cooled MCT detector. The sampling apparatus was a Minidiff diffuse reflectance unit with SELECTOR *x–y* translational stage with manual vernier controls (SPECAC Ltd., Kent, UK). The instrument was interfaced to a personal computer running Mattson's WINFIRST software for instrument control, data acquisition, and analysis. DRIFTS data presented herein were collected under the following instrumental conditions: Happ–Genzel apodization, 2× zero fill, forward scanning velocity of 20 kHz, backward scanning velocity of 20 kHz, resolution of 8 cm^{-1}, 256 co-added scans, and scanning range of 400 cm^{-1} to 4000 cm^{-1}. For quantitation, background infrared spectra were obtained on a blank glass fiber filter and these were automatically subtracted from the collected spectra of samples. In addition, energy throughput from diffuse reflectance of the samples was measured at the beginning and end of scanning, and the sample results were then normalized by dividing by the average throughput value, typically in the range of 3 V. This procedure served to even out day-to-day and sample-to-sample variations in both instrument performance and sample reflectivity.

Several modifications were made to the Minidiff diffuse reflectance apparatus to enhance its applicability to analysis of Respicon™ filter samples. The diffuse reflectance stage was modified to ensure that the filter samples were reproducibly placed in position and could be scanned using pre-set marks on the stage verniers. The stage was modified to accommodate a standard 37 mm polystyrene filter cassette bottom (SKC, Inc., Eighty Four, PA) as the actual filter sample holder. A mounting tube was attached to the stage that dovetailed with the bottom filter cassette inlet, thus ensuring that the cassette/sample holder was always in the same position on the sampling stage. The other end of the mounting tube was attached *via* latex tubing to a diaphragm vacuum pump which continuously applied suction to the sample during infrared scanning, thus holding it perfectly flat and in position. The cassette bottom/filter holder was also modified by removing the upper rim, except for 3 tabs that acted to position the filter in the holder. This was done in order to scan the filter all the way to its edges without the cassette rim interfering with the infrared beam. The modified cassette filter holder was painted matte black to minimize scattered radiation. A schematic of the sample holder is shown in Fig. 2.

Visual inspection and preliminary DRIFTS scans of actual Respicon™ sample filters collected in the field indicated that the dust on the Respicon™ filters is not uniformly distributed across the filter surface. Since the scan area of the infrared beam is small in comparison to the filter area, a large number (>100) of stationary area scans on a single filter sample would be required to obtain a reliable average response for accurately quantitating the dust on a filter. In order to reduce the number of scans required for accurate evaluation of the samples, the diffuse reflectance device was further modified so that integrated analysis of a large area of the filter could be obtained in a single scanning period. The modification consisted of a clockwork motor (Synchron 600, Hansen Motor Co., Princeton, IN) coupled to the horizontal *x–y* stage vernier which slowly moved the stage, and thus the scanning point on the filter, at a constant rate in the horizontal direction. The drive motor was reversible and was controlled by a three-way switch so that the filter sample could be scanned in either horizontal direction conveniently. The angular velocity of the motor (4 rev min^{-1}) was chosen to ensure synchronization of the stage scanning time with the time required for collection of the infrared spectra, approximately 90 s. In practice, a scan was taken of the filter sample in the horizontal direction and subsequently, the filter holder was rotated 90° and a second horizontal scan of the filter sample was taken. Thus in comparison to stationary analysis of a sample, this modification reduced the number of required DRIFTS scans per sample to just two, and the analysis time from several hours to several minutes.

Standard wood materials for use in preparation of analytical reference standards were obtained from a local retail lumber store. Kiln-dried, dimensional boards or dowels of southern yellow pine, Radiata pine, red oak, maple, birch, cherry, and poplar were purchased for this purpose. Size-fractionated dusts of the reference woods were generated and collected using a special apparatus in the laboratory. A disc/belt sander (Delta Machine Co., Jackson, TN) was placed inside a small bench-top polypropylene laboratory hood (Lab Safety Supply Co.). The sander was fitted with a 125 mm diameter sanding disc, medium grit abrasive, (Norton Abrasives, Niagara Falls, NY). A slot was cut into the side of the hood through which the wood board or dowel could be passed and fed into the sander. The sash of the hood was lowered and dust was generated by manually feeding the board into the disc sander.

A Respicon™ sampler (TSI Instruments, St. Paul, MN) was placed inside the hood during sanding and used to collect dust to be used as a reference standard. For this purpose, the Respicon™ was loaded with 37 mm diameter, 2 μm pore size Teflon filters in each of its three stages, and operated at its nominal flow rate of 3.1 l min^{-1}. After sampling, the collected dust cakes were carefully scraped from the surface of the filters and transferred to sample vials. The collected wood dust was then placed in a vacuum desiccator for drying and storage. This procedure resulted in the collection of reference wood dusts for each species that were fractionated into three size ranges corresponding to the Respicon™ stages: stage 1, respirable particles; stage 2, tracheobronchial particles; and stage 3,

extra-thoracic particles. These size fractions are defined in accordance with the ACGIH/ISO/CEN criteria for size-selective sampling of airborne particles.[8]

Analytical standards were prepared by weighing out an appropriate amount of the dried, size-fractionated reference dust and then suspending it in a known amount of ethyl acetate to produce a standard dust suspension. Ethyl acetate was chosen for this procedure because of its low polarity, low viscosity, and high dielectric constant; these properties prevent emulsification and enhance the filtration of suspended wood particles. Exact aliquots of the standard suspension were then taken and filtered onto the same 37 mm glass fiber filters (Omega Specialties, Chelmsford, MA) that were normally used in field sampling with the Respicon™ sampler. The filters were mounted in a special filtration apparatus made from the bottom piece and five middle rings obtained from 3-piece solvent resistant 37 mm filter cassettes (SKC, Inc., Eighty Four, PA). The filter was placed onto a back-up glass fiber filter and friction sealed into the filter apparatus between the bottom piece and the lower of the middle rings. Approximately 30 ml ethyl acetate was placed on top of the filter, the standard aliquot was then added and stirred into the solvent, and vacuum was applied to the filtration apparatus. The wood dust standard (primary and back-up filters) was then removed from the apparatus and vacuum dried prior to use.

For preparation of standards for respirable and tracheobronchial dusts, ''holed'' glass fiber filters identical to those used in Respicon™ stages 1 and 2 were used. In these cases, a 7.5 mm disc of glass fiber filter material, slightly larger than the center hole in the filter, was placed under the hole and in between the primary and back-up filters to prevent loss of dust onto the back-up filter, and to provide a more uniform plane of analysis for the diffuse reflectance measurements.

Respicon™ filter samples collected in the wood processing industry exhibited a very heterogeneous surface distribution of dust, with high concentrations of dust localized around the Respicon™ filter's center hole. Preliminary DRIFTS analyses of such samples yielded several observations: the DRIFTS spectra showed many overlapping absorbance bands in comparison to standards prepared by filtering aliquots of dust suspended in solvent; the energy throughput was severely compromised when the infrared scanning beam was located directly on top of localized areas of thick dust cake on the filter surfaces; and the DRIFTS response to increasing mass of dust was asymptotic with a narrow range of dust mass before saturation of the signal.

In order to obviate these problems, samples were pre-treated as follows: the dust-laden Respicon™ filter was mounted in the solvent-resistant filtration apparatus on top of a back-up glass fiber filter, and approximately 30 ml ethyl acetate was poured on top of the sample filter. The dust cake was then dispersed into the ethyl acetate by gentle stirring of the liquid; subsequently, vacuum was applied to the filtration apparatus and the suspended dust was re-deposited onto the filter in a uniform pattern. For ''holed'' filter samples obtained from Respicon™ stages 1 and 2, the previously described procedure of placing a glass fiber disc under the hole prior to solvent re-suspension was followed. During the vacuum filtration of such ''holed'' filters, a 7.5 mm diameter piston was placed on top of the hole/disc and held firmly in place to prevent leakage of particles around the perimeter of the hole. This pre-treatment protocol not only resulted in a more homogeneous surface deposition/filter cake, but also provided a solvent wash to help eliminate interfering infrared bands arising from natural wood extractables and non-wood particulate matter such as spray-finish aerosol and environmental tobacco smoke.

In order to examine possible differences in infrared response across wood species, standards were prepared from wood dust obtained from sanding of Radiata pine, poplar, cherry, maple, and birch in addition to southern yellow pine and red oak. These species were chosen based on their being used with some frequency in the ten facilities under study in a companion project evaluating respiratory health of industrial wood workers. For each of these species, five replicate 1.0 mg standards were prepared from extrathoracic sanding dust and analyzed by DRIFTS with evaluation at both 1251 and 1291 cm^{-1}.

Figures of merit, including precision and measurement range for the analytical technique, were determined from multiple analyses of 18 standards prepared from the size-fractionated, reference southern yellow pine sanding dust. Six standards, ranging from 250 μg to 2 mg of dust per filter, were prepared for each of the size fractions. Each standard was analyzed 7 times and the results combined to determine figures of merit. Standard curves resulting from quantitation using ''normalized net absorbance'' were developed. The ''net absorbance'' was obtained as the diffuse reflectance value (Kubelka Munk Units, KMu) at 1251 cm^{-1}, and normalized by dividing by the average energy throughput value (volts) of the standard. The precision of the method was taken as the pooled relative standard deviation (coefficient of variation) for multiple analyses of the standards.

The limit of detection (LOD) was determined according to the protocol suggested by Taylor.[9] The variability of the measurement as sample amount approaches zero (s_0) was estimated by the observed standard deviation of analysis of a standard in an amount near the expected LOD. The LOD was then taken as the equivalent of $3s_0$, resulting in a 95% confidence interval of $\pm 100\%$ at the LOD.

The performance of the method was evaluated by analyzing a total of 51 Respicon™ filter samples collected from test atmospheres of southern yellow pine sanding dust generated in the laboratory. These samples were collected by Respicon™ samplers loaded with pre-weighed glass fiber filters for various periods of time to yield a range of collected dust weights for each of the size fractions. After collection, the filter samples were removed from the Respicon™ and vacuum dried prior to re-weighing to determine gravimetric loading of dust. The samples were then subjected to the ethyl acetate re-suspension/re-filtration technique and submitted for DRIFTS analysis in comparison to the appropriate size-fractionated southern yellow pine sanding dust reference standards. The results of this performance test were analyzed using a multiple linear regression model with gravimetrically determined mass of dust as the dependent variable, and independent variables for mass determined by DRIFTS, and interaction terms with mass by DRIFTS and dummy variables for particle size fraction (corresponding to the respective Respicon™ stage).

Selected materials were evaluated as potential interferants in the DRIFTS determination of wood dust. These included rubber, environmental tobacco smoke, and spray-on wood finishes. Rubber particulate matter was obtained as shavings from a black latex rubber laboratory stopper. These shavings were placed in an analytical mill with dry ice and reduced in size to a fine powder. A portion of rubber powder ($\sim$12 mg) was weighed out and then suspended in ethyl acetate and filtered onto a glass fiber filter. Environmental tobacco smoke (ETS) was generated from a commercial brand of cigarette that was smoked in a small bench-top smoking apparatus. The sidestream smoke from the burning cigarette was captured by air sampling onto a glass fiber filter. The glass fiber filter containing approximately 6 mg of ETS was washed with ethyl acetate prior to DRIFTS analysis. The spray-on finishes included a polyurethane, Olympic Interior Oil-Based Polyurethane (PPG Architectural, Inc., Pittsburgh, PA) and two acrylics, American Tradition Ultra Clear Acrylic (Valspar, Wheeling, IL) and Rust-Oleum Specialty Lacquer (Rustoleum Corp., Vernon Hills, IL). Overspray aerosol from each of the finishes was generated by spraying inside a laminar-flow aerosol test chamber and then sampling onto 37 mm glass fiber filters. In each case, sample time was sufficient to collect

approximately 1 mg of overspray aerosol. The overspray samples were then washed with ethyl acetate and submitted for DRIFTS analysis.

Simple statistical analyses of the data such as descriptive statistics and linear regressions were performed using Microsoft Excel 2000. More complex analyses including 2-way ANOVA, *post hoc* pairwise comparisons, and multiple regressions, were done with Sigmastat 3.0 software (SPSS Inc., Chicago, IL).

Results and discussion

The diffuse reflectance infrared spectra of wood species and cellulose all showed a strong doublet with maxima at approximately 1251 and 1291 cm^{-1} (Fig. 1). Our primary focus was on southern yellow pine and red oak as these two species were the most commonly used in our survey of the American wood processing industry. In red oak, the two maxima were approximately equal in magnitude, whereas in southern yellow pine, the maximum at 1251 cm^{-1} was approximately twice the magnitude of that at 1291 cm^{-1}. Furthermore, the 1291 cm^{-1} peak appeared to exhibit roughly equivalent response for both oak and pine. Because our primary objective was to develop a method that could quantitate total wood dust, without regard to the relative amount of softwood and hardwood in the samples, we focused our development on using the two observed absorbance maxima in separate univariate calibrations, rather than either combinations of these or other wavelengths or integrated absorbance over the entire absorbance band of 1200–1300 cm^{-1}.

In addition to red oak and southern yellow pine, commonly encountered species in American wood processing industries include Radiata pine, and the hardwoods poplar, cherry, birch, and maple. The results of DRIFTS analysis of all seven different species of wood are shown in Table 1. The data in Table 1 were analyzed using a two-way ANOVA with species and wavenumber as the treatment variables. There were statistically significant differences in response among the species of wood for both wavenumbers, as well as significant interaction between species and wavenumber. All pair-wise combinations were tested for significance using the Student–Newman–Keuls procedure. At 1251 cm^{-1}, there were many significant pairwise differences in response across the species of wood. At 1291 cm^{-1}, maple was found to be significantly different from all the other wood species, but none of the remaining species

Table 1 Comparison of DRIFTS response of seven species of wood

Species (1.0 mg each)	Normalized net absorbance/KMu V^{-1} (mean ± standard error)	
	1251 cm^{-1}	1291 cm^{-1}
Southern yellow pine	0.529 ± 0.018	0.234 ± 0.013
Radiata pine	0.362 ± 0.018	0.188 ± 0.019
Red oak	0.226 ± 0.018	0.220 ± 0.013
Poplar	0.260 ± 0.016	0.219 ± 0.016
Cherry	0.272 ± 0.014	0.254 ± 0.014
Maple	0.089 ± 0.012	0.098 ± 0.016
Birch	0.297 ± 0.016	0.213 ± 0.016

Two-way analysis of variance

Source	DF	SS	MS	*F*	*p*
Species	6	0.783	0.131	66.683	<0.001
Wavenumber	1	0.193	0.193	98.749	<0.001
Species × wavenumber	6	0.301	0.0502	25.656	<0.001
Residual	108	0.211	0.00196		
Total	121	1.334	0.0110		

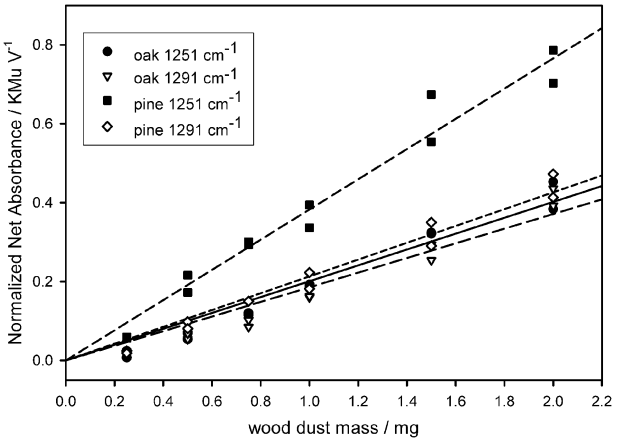

Fig. 3 DRIFTS standards for extrathoracic pine and oak dusts at 1251 cm^{-1} and 1291 cm^{-1}.

was significantly different from the others. It is uncertain why maple exhibited such a significantly lower response in comparison to DRIFTS of the other species. However, it was noted that during the generation of the reference dusts, maple tended to scorch and char during machine sanding, presumably because of its extreme hardness. It may be that there was significant thermal degradation of its cellulose content during this procedure.

Overall, these results indicate that, with the exception of maple, determination of wood dust can be done regardless of species with quantitation using the 1291 cm^{-1} peak. Use of this peak results in diminished sensitivity for some species, such as southern yellow and Radiata pines; however in those situations where there is clearly only one species present, quantitation may be done with the appropriate standard at either wavenumber for enhanced sensitivity. For illustration, Fig. 3 shows standard curves for extrathoracic dusts from red oak and southern yellow pine measured at both wavenumbers. The three lower standard curves representing pine at 1291 cm^{-1}, oak at 1251 cm^{-1} and oak at 1291 cm^{-1} are not statistically different.

DRIFTS analysis of particulate samples is known to be affected by particle size distribution, with response generally increasing as particle size decreases. In light of this, we compared the response of wood dust collected from each of the three size fractions corresponding to stages of the Respicon™ sampler. Standards of southern yellow pine sanding dust from each Respicon™ stage, ranging from approximately 200 μg to 2 mg of dust were prepared and analyzed for this purpose. Multiple linear regression analysis was performed using normalized net absorbance at 1251 cm^{-1} as the dependent variable, mass of dust as the primary independent variable, and with interaction terms for mass and dummy variables for particle size fraction. The results are shown in Table 2.

The regression model indicated highly significant differences in response among dusts from the three stages. Taken separately, the response factors (slopes) for the three standard curves were 0.333, 0.259, and 0.407 KMu V^{-1} mg^{-1}, for respirable, tracheobronchial, and extrathoracic dusts, respectively. These results dictate that separate standards should be used for analysis of samples from the three Respicon™ stages and that particulate materials analyzed by DRIFTS should employ standards that are matched to the samples in terms of particle size distribution.

The precision of analysis was similar for all three particle size standards. The pooled coefficients of variation for the three sets of southern yellow pine standards, analyzed using the 1251 cm^{-1} peak, were 5.5%, 7.4%, and 5.4% for respirable, tracheobronchial, and extrathoracic dusts, respectively. For individual standards, the coefficients of variation ranged from

Table 2 Multiple regression model for DRIFTS analysis of size-fractionated pine dust standards

Independent factor	Coefficient	*p*-Value
Mass/mg	0.407	<0.001
Mass × respirable size fraction (Respicon™ stage 1)	-7.39×10^{-2}	<0.001
Mass × tracheobronchial size fraction (Respicon™ stage 2)	−0.148	<0.001

Dependent variable = normalized net absorbance at 1251 cm^{-1} (KMu V^{-1}).

Table 3 Assessment of analytical interferences (post-ethyl acetate treatment) in the DRIFTS determination of wood dust

	Equivalent mg southern yellow pine per mg of material	
Material	1251 cm^{-1}	1291 cm^{-1}
Rubber	0.025	0.016
Environmental tobacco smoke	None	None
Acrylic lacquer	None	None
Acrylic gloss	None	None
Polyurethane	0.39	0.36

1.2% to 12.8%. In general, standards in the lowest ranges (250–500 μg) exhibited the largest variability, and the coefficients of variation tended to diminish as the amount of dust increased. Overall, the pooled coefficients of variation were quite consistent, regardless of the size fraction being analyzed. The overall pooled coefficient of variation was 6% which is within a desirable maximum variability of ~10% for analytical techniques used in support of workplace exposure assessments.

The upper range of measurement of the technique is approximately 4 mg of dust per sample, depending upon the particular size fraction being analyzed. At this upper limit, the diffuse reflectance begins to exhibit signs of saturation and the analytical response becomes non-linear and asymptotic. The lower range of measurement is equal to the analytical limit of detection (LOD) of the technique. Within the constraints of potential day-to-day instrument variability, a LOD of about 80 μg of pine sanding dust is a reasonable average estimate.

The comparison between gravimetric and DRIFTS analysis of pine sanding dusts exhibited a very high degree of correlation and consistency. The multiple regression model indicated there was no statistically significant difference in the results of this comparison across the three size fractions of dust. However, the model also suggested that there was a statistical difference, albeit small in magnitude, between the DRIFTS and gravimetric analyses. To examine this further, the non-significant interaction terms were eliminated from the statistical model and a simple linear regression was run on the results with dust mass by DRIFT spectroscopy as the dependent variable and the gravimetric mass as the independent variable. The entire dataset and the resulting regression line are shown in Fig. 4.

The regression analysis of the data showed that the mass of pine sanding dust determined by DRIFTS was statistically different from that by gravimetric analysis of the same samples. Overall, the DRIFTS results were approximately 8% higher than the respective gravimetric data. A common target for the maximum tolerable bias in analytical techniques used for exposure assessment in industrial hygiene is ±10%, and the data were within that value. However, it should be pointed out that these data incorporated a certain unknown amount of error in the gravimetric results themselves, primarily as a result of the unavoidable loss of friable filter material from loading and unloading of these samples from the Respicon™ sampler. Such error would result in an underestimation of the actual gravimetric weight. As such, it is likely that the true difference between the DRIFTS and gravimetric results is somewhat less than 8%.

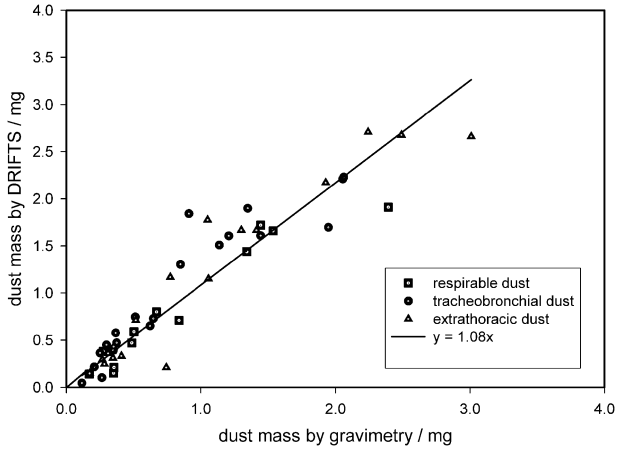

Fig. 4 Comparison of Respicon™ samples of airborne pine sanding dust analyzed by gravimetry and by DRIFTS.

Table 3 presents the results of the evaluation of the potential analytical interferants. Environmental tobacco smoke and the acrylic finishes showed no response at either 1251 or 1291 cm^{-1} and therefore are not interferences for the method. It should be noted that DRIFTS screening of these samples prior to ethyl acetate treatment did indicate some activity at the wavenumbers of interest; however, after ethyl acetate washing, the infrared response was completely eliminated. Rubber showed minimal response under the analytical conditions, with an interference level equal to 2.5% and 1.6% of that of wood dust when measured at 1251 and 1291 cm^{-1}, respectively. The most significant of these interferants was the polyurethane spray-on finish, which presented an interference level of 36 to 39% of that of wood dust. It was observed that the polyurethane material was also somewhat resistant to the ethyl acetate wash, as the DRIFTS scans collected pre-/post-ethyl acetate wash indicated a reduction of only about half of the initial infrared response of this material.

Conclusions

The DRIFTS technique is suitable for the determination of wood dust in size-fractionated samples of airborne particulate collected from the wood processing industry. This technique results in a specific measurement of wood in these samples and is largely unaffected by the presence of other potential particulate materials such as environmental tobacco smoke and spray finish aerosols. Quantitation of wood using the infrared response at 1291 cm^{-1} is appropriate for unbiased measurement of wood species commonly used in North America except for maple, which exhibited a significantly lower infrared response in comparison to six other commonly used species. Reference wood dusts produced for use as analytical standards should be matched to the expected size distributions of collected dust samples because of a difference in infrared response with particle size distribution. The DRIFTS method is applicable for determination of wood dust loadings in the range of about 0.1 to 4 mg per filter sample. This technique is a significant improvement in the determination of wood dust in comparison to other approaches that have relied upon the analysis of minor constituents of wood, such as terpenes and tannins, as surrogates for whole wood dust.

Acknowledgements

The technical assistance of Zachariah Fridge and Latonya Senegal is greatly appreciated. This work was supported by the American Forest & Paper Association.

References

1 International Agency for Research on Cancer Working Group, *Wood dust. IARC Monographs on the Evaluation of Carcinogenic Risks of Chemicals to Humans*, IARC, Lyon, 1995, vol. 62.
2 American Conference of Governmental Industrial Hygienists (ACGIH), *Threshold Limit Values for Chemical Substances and Physical Agents and Biological Exposure Indices*, ACGIH, Cincinnati, 2004.
3 P. Mämmelä, A. Tuomainen, H. Savolainen, J. Kangas, T. Vartiainen and L. Lindross, *J. Environ. Monit.*, 2001, **3**, 509.
4 K. Teschke, P. Demers, H. Davies, S. Kennedy, S. Marion and V. Leung, *Ann. Occup. Hyg.*, 1999, **43**, 247.
5 P. Demers, K. Teschke, H. Davies, S. Kennedy and V. Leung, *Am. Ind. Hyg. Assoc. J.*, 2000, **61**, 521.
6 G. A. Smook, *Handbook for Pulp and Paper Technologists*, Technical Association of the Pulp and Paper Industries, Atlanta, 1982, p. 15.
7 R. J. Rando, H. G. Poovey, D. Mokadam, J. Brisolara and H. Glindmeyer, *J. Occup. Environ. Hyg.*, 2005, **2**, 219.
8 International Organization for Standardization, *Air Quality—Particle Size Fraction Definitions for Health-Related Sampling*, ISO, Geneva, 1991.
9 J. K. Taylor, *Quality Assurance of Chemical Measurements*, Lewis Publishers, Chelsea, MI, 1987, pp. 79–82.