ON TRIMODAL PARTICLE SIZE DISTRIBUTIONS IN FLY ASH FROM PULVERIZED-COAL COMBUSTION

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Combustion-generated fine particles, defined as those with aerodynamic diameters less than 2.5 \( \mu m \), have come under increased regulatory scrutiny because of suspected links to adverse human health effects. Whereas classical theories regarding coal combustion suggest that mechanisms of ash vaporization and fragmentation lead to bimodal ash particle size distributions (PSDs), this paper presents experimental results supporting other existing hypotheses that three distinct ash modes may be more appropriate. This paper focuses on the existence and generality of a central mode, between approximately 0.7 and 3.0 \( \mu m \) diameter. This central mode is presumably caused by fragmentation mechanisms, but is still important from a health perspective, because a large portion is contained within the 2.5 \( \mu m \) particle size fraction. Presented here are experimental results from two different laboratory combustors and one industrial boiler, all burning pulverized coals. Use of a variety of particle-sampling and size classification methods, including electrical mobility, time-of-flight, and inertial (low-pressure impaction) methods, confirms that the central mode is not an artifact of the particle-sampling and -sizing methods used. Results from the combustion of 10 different coals consistently show that this central mode is significant for both high- and low-rank coals. Size-segregated elemental distributions of calcium, iron, and aluminum provide additional insight into mechanisms of formation of each mode. Field tests show that the central mode can be the major contributor to fine particle emissions leaving an electrostatic precipitator (ESP). The new experimental results presented here are interpreted in the light of complementary existing data and available theories from the literature.

Introduction

Airborne fine particulate matter (PM) has been the subject of considerable recent environmental interest as a result of a number of research studies correlating short-term exposure of ambient levels of fine PM with acute adverse health effects [1]. These studies were summarized by the Environmental Protection Agency (EPA) [2] and reviewed by EPA's Clean Air Scientific Advisory Committee, which concluded that there was evidence linking ambient fine PM concentrations and adverse health effects [3]. These studies were the basis for a revision of the National Ambient Air Quality Standards for PM that included a standard for PM less than 2.5 \( \mu m \) in aerodynamic diameter (PM\(_{2.5}\)) [4].

Combustion of pulverized coal leads to emission of fly ash particles containing iron and other transition metals which have been identified as possible causative agents for adverse health effects [5]. Furthermore, a significant fraction of coal fly ash mass is contained in particles less than 2.5 \( \mu m \) in aerodynamic diameter. Sarofim et al. [6] and Flagan and Friedlander [7] describe how particles from pulverized-coal combustion are formed through at least two mechanisms, fragmentation and vaporization, resulting in a bimodal fly ash particle size distribution (PSD). The field data of McElroy et al. [8] support this finding, showing an ultrafine ash vaporization mode centered at approximately 0.1 \( \mu m \) diameter and a coarse fragmentation mode apparently centered at approximately 20 \( \mu m \) diameter.
These data also show that the ultrafine particle mode becomes more pronounced downstream of particulate collection devices, such as an electrostatic precipitator (ESP), due to preferential removal of the large coarse mode PM. Kauppinen and Pakkanen [9] also present combustion aerosol PSDs measured after an ESP, showing two modes centered at approximately 0.05 and 2 μm diameter. They reported that larger particles were effectively removed by the ESP.

More recent results [10–12] suggest that pulverized-coal combustion will yield three, rather than two, primary ash aerosol size modes. These include a vaporization mode centered at approximately 0.08 μm diameter and two fragmentation modes centered at approximately 2 and 20 μm diameter. The middle mode, or fine fragmentation mode, has been attributed to ash formation from char cenospheres [11–13] or to ash ejection from rapidly rotating char particles [10]. Prior to this, Smith et al. [14] had also observed two fragmentation modes. One was a fine fragmentation mode due to bursting of hollow ash spheres, and a coarse fragmentation mode due to coalesced ash formed during char burnout. However, because of its inherently small mass, Smith et al. [14] did not emphasize the additional importance of the ultrafine vaporization mode.

The results provided in the present work highlight the ubiquitous nature of the fine fragmentation mode. This is an important issue for those desiring to determine mechanisms governing the composition, mass, and ensuing health effects of coal combustion PM, a significant fraction of which reside within this fine fragmentation mode.

**Experimental**

**Combustors**

Data are presented from two down-fired laboratory-scale combustors (4 and 2.2 kg/h) and one field-scale pulverized-coal-fired boiler (63.5 tonnes/h). One laboratory-scale combustor (EPA combustor) consists of a vertical 4.1 m long, 20 cm i.d. refractory-lined vertical tube. Details are described elsewhere [15]. Pulverized coal (4 kg/h) was metered from a screw feeder and carried by transport air through a fuel injector into the combustor. Additional axial and tangential air streams were metered separately through the variable-swirl burner as annular flow around the coal. Following the vertical section, the combustion gases turn into a 3.7 m long, 15 cm i.d. horizontal duct. Ports are available along the furnace and exhaust duct for extractive sampling.

The other laboratory-scale combustor (University of Arizona [UA] combustor) is 15 cm i.d. and 6 m long, consisting of a slag-resistant refractory-lined section followed by a fibrous alumina section, both insulated by a Kaowool blanket. Pulverized coal (2.2 kg/h), transported by primary air, was mixed with secondary air prior to combustion, forming an essentially "premixed" pulverized-coal flame. For both the EPA and the UA combustors, the resultant temperature profiles were those occurring naturally without additional heating. Both furnaces were designed to replicate the time/temperature histories and coal and ash particle concentrations present in practical units.

The full-scale pulverized-coal boiler (Ishikawajima-Harima Heavy Industries [IHI]-tested facility) is an industrial boiler with a steam rating of 550 tonnes/h. This facility, operated at 510 tonnes/h of steam (coal feed rate of 63.5 tonnes/h), is equipped with a catalytic DeNOx reactor, followed by an air heater, ESP, induced draft fan (IDF), and flue gas desulfurization (FGD) system. Data are presented from sampling locations before and after the ESP at 139 and 136 °C, respectively, and after the FGD system (stack inlet).

**Coal Compositions**

Table 1 presents the proximate and ultimate analyses for the ten coals investigated in this study. There were six bituminous coals, two subbituminous coals, one lignite, and one bituminous/subbituminous coal blend. One might expect coal rank to be important if swelling behavior were important in determining the shape of the coal ash PSD. To allow comparison between laboratory- and full-scale PM emissions, total stack particulate concentrations are given for the seven coals tested in the EPA combustor (prior to particle control) [16]. Similar measurements are presented for the IHI-tested facility at the ESP inlet, ESP outlet, and stack inlet. All coals were burned under excess air conditions (3%–4% stack oxygen). Where measurements exist, unburned carbon in the EPA facility ash ranged from 0.5% to 10% for a subbituminous and two bituminous coals, respectively. Similar measurements for the IHI-tested facility ash ranged from 1% to 3%. Ash carbon values for the UA combustor are likely similar to the EPA values, since combustion conditions were similar.

**Sampling and Particle Size Measurements**

PSDs reported here were determined by a combination of three techniques. Instruments based on electrical mobility (scanning mobility particle sizer, SMPS), time-of-flight (aerodynamic particle sizer, APS), and inertial (low-pressure impaction, LPI) measurements were used for extracted aerosols. Extractive samples were collected using isokinetic aerosol-sampling systems described elsewhere [17,18]. For the EPA combustor, these samples were diluted and directed to TSI Inc. SMPS and APS instruments. The SMPS and APS were configured to
### TABLE 1

Coal analysis

<table>
<thead>
<tr>
<th>Pittsburgh no. 8</th>
<th>Western no. 8</th>
<th>Wisconsin</th>
<th>Wyoming PRB</th>
<th>North Dakota Subbit</th>
<th>Ohio Blend</th>
<th>Pittsburgh PRB</th>
<th>Illinois no. 6 Bit</th>
<th>Eastern Kentucky Bit</th>
<th>Indonesia/Australia Blend</th>
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<tr>
<td>Moisture (%)</td>
<td>2.67</td>
<td>6.97</td>
<td>11.36</td>
<td>5.97</td>
<td>25.81</td>
<td>35.88</td>
<td>2.33</td>
<td>3.31</td>
<td>2.33</td>
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<tr>
<td>Ash (%)</td>
<td>8.65</td>
<td>7.51</td>
<td>10.41</td>
<td>9.69</td>
<td>6.03</td>
<td>9.38</td>
<td>9.70</td>
<td>7.01</td>
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#### Proximate Analysis

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<td>25.81</td>
<td>35.88</td>
<td>2.33</td>
<td>3.31</td>
<td>2.33</td>
<td>2.7</td>
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<td>Fixed carbon</td>
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<td>49.66</td>
<td>41.05</td>
<td>45.75</td>
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<td>26.59</td>
<td>48.78</td>
<td>61.99</td>
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<tr>
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<td>7.51</td>
<td>10.41</td>
<td>9.69</td>
<td>6.03</td>
<td>9.38</td>
<td>9.70</td>
<td>7.01</td>
<td>10.26</td>
<td>7.41</td>
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<tr>
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<td>4927</td>
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<td>4927</td>
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#### Ultimate analysis

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<thead>
<tr>
<th>C</th>
<th>74.51</th>
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<th>64.87</th>
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<td>O</td>
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<td>17.56</td>
<td>13.18</td>
<td>16.52</td>
<td>19.52</td>
<td>8.28</td>
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<td>11.74</td>
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<td>8.13</td>
<td>14.36</td>
<td>9.92</td>
<td>7.01</td>
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#### PM conc. (mg/m³)

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<tr>
<th>(std. dev.)</th>
<th>3565</th>
<th>3807</th>
<th>4374</th>
<th>4323</th>
<th>3441</th>
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<th>4477</th>
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<td></td>
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<tr>
<td>Unburned carbon, (wt %)</td>
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<tr>
<td>LOI (wt %)</td>
<td>12.9</td>
<td>2.3</td>
<td>14.5</td>
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</tbody>
</table>

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*a* As received (wet).

*b* The IHI tested coal was a 50/50 (by weight) blend of an Australian subbituminous and an Indonesian bituminous coals.

*c* Higher heating value.

*d* Dry basis.

*e* By difference.

*f* These values represent measured PM emissions at the ESP inlet, ESP outlet, and stack inlet, respectively.

*g* Determined at electrostatic precipitator inlet.

*h* Loss on ignition.

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yield 54 and 50 channels, respectively, evenly spaced (logarithmically) over 0.01–1.0 and 0.5–20 µm diameter ranges, respectively.

Particles were sampled from the UA combustor in a similar manner [19]. Diluted samples were routed through polyethylene tubing to a Berner design LPI [20]. This LPI controls the flow rate through a critical flow orifice and classifies sampled particles onto 11 stages. To collect sufficient mass of ultrafine particles for gravimetric measurements, the LPI was operated in two modes. First, a cyclone was attached to the inlet to remove particles (>1 µm) that would normally be collected on stages 7 through 11. This allowed sampling times to be long enough (~5–30 min) to collect measurable masses of ultrafine particles on the lower stages, without complications caused by large particle bounce. Then, the cyclone was removed and the LPI was operated for a short time (~0.5–2 min) which was insufficient to obtain measurable masses on the lower stages, but which allowed a PSD to be obtained for the larger particles collected on the upper stages. The PSD for the entire particle size range was obtained by combining results from both LPI operating modes. Each run consisted of at least three replicate sets of LPI samples. In addition to gravimetric measurements, impactor samples were analyzed using atomic absorption and graphite furnace atomic absorption spectroscopy.

For the IHI-tested pulverized-coal boiler, samples were withdrawn at three locations: (1) before the ESP but after the DeNOₓ system and the air heater, (2) after the ESP but before the IDF, and (3) before the stack inlet but after the FGD system. Particles were segregated using an LPI with 12 stages and an afterfilter. In contrast to the UA procedure, the IHI samples were not diluted, and the LPI assembly was maintained at 130 °C to prevent moisture condensation. For purposes of presentation here, the lower
Fig. 1. Fly ash PSDs for seven coals as measured by SMPS and APS.

Results

Figure 1 presents fly ash PSDs for seven coals (see Table 1) burned in the EPA combustor. PSDs for high- and low-rank coals are shown on the upper and lower panels, respectively. Again, note that these data are derived from measurements from two instruments (SMPS and APS). The PSDs agree remarkably well in the region where they overlap. The combined range covers the evolving ultrafine nuclei mode as well as both fine and coarse fragmentation modes up to 20 μm diameter. Particles larger than 20 μm diameter are outside the APS range. The data clearly show three modes for all coals: a small ultrafine mode at approximately 0.1 μm diameter and two larger modes presumably the result of fragmentation processes. These observations are consistent with findings by others [10–14]. The central mode is centered at approximately 1–2 μm diameter, and the edge of the coarse mode is shown rising at approximately 10 μm diameter. The fact that there are two distinct modes larger than 0.5 μm diameter, rather than a single broad fragmentation mode, is significant and suggests two distinct fragmentation mechanisms, for both high- and low-rank coals. As expected, the North Dakota lignite and Wyodak Powder River Basin (PRB) coals have the larger nuclei modes, most likely due to higher alkali metal ash contents [21].

Figure 2 presents PSDs for six coals burned in the UA combustor, plotting differential fraction of the total mass collected on LPI stages versus the (lower) particle aerodynamic cut-off diameter. Of these six coals, three (Ohio blend, Wyodak, North Dakota) are identical to those shown on Fig. 1. Whereas the PSDs on Fig. 1 were obtained from SMPS and APS instruments, the PSDs on Fig. 2 were obtained from gravimetric determination of fly ash samples segregated and collected using an LPI. Even though these LPI measurements are less resolved, there are distinct central modes at approximately 2 μm diameter for the Ohio, Wyodak, Pittsburgh, and Illinois coals, and strong inflection points in the same region (suggesting center modes) for the North Dakota and Eastern Kentucky coals. It should be noted that both high- (Ohio, Illinois) and low-rank (Wyodak) coals show distinct central modes, indicating a particle formation mechanism (or mechanisms) that is not directly dependent on coal rank.

Size-segregated aerosol compositions of the major elements, silicon, aluminum, iron, calcium, magnesium, and sodium (reported as oxides), are also
Fig. 3. Fly ash PSDs from a full-scale industrial boiler as measured by LPI before and after an ESP and also after an FGD unit (stack inlet).

shown on Fig. 2 for the Ohio and Wyodak coal ash samples representing each of the three modes. Particles representing the ultracine/vaporization mode were collected on stage 3. Particles representing the central particle mode were collected on stage 8 at an aerodynamic particle diameter of 1.96 μm. Particles representing the larger coarse mode particles were collected on stage 11. With slight variations, the composition data for particles on stage 8 are similar to those for the larger sizes (stage 11), and very different from the ultrafine particles (stage 3). The ultrafine particles contain notably smaller quantities of silicon and aluminum compared with concentrations on stages 8 and 11. This supports the characterization of the central mode as a “fine fragmentation mode,” and is in agreement with Smith et al. [14]. Note that the ultrafine particle modes for both coals contain relatively large concentrations of iron and sodium. Calcium comprises a significant fraction of the ultracine particle mode of the Wyodak coal. Certain trace and toxic metals such as arsenic and selenium will combine with and track calcium and/or iron. [19,22].

Field data from the IHI-tested facility are depicted on Fig. 3. The upper figure shows the ash aerosol PSD entering the ESP, while the lower panel shows the ash PSDs leaving the ESP and the FGD system (stack inlet). The PSD on the upper panel shows a definite inflection point at approximately 2 μm diameter on the shoulder of the large coarse mode. The PSD of the aerosol exiting the ESP shows a definite fine fragmentation mode at approximately 1 μm diameter because of the high removal efficiency of the larger particles. In fact, the fine fragmentation mode persists through the acid gas scrubber, where it appears to be joined by yet another (possibly acid aerosol) mode. Both modes are ultimately emitted to the atmosphere. A distinct ultracine mode at approximately 0.1 μm diameter is also evident from both PSDs on the lower panel (after the ESP). These data confirm that the fine fragmentation mode is of practical importance and is a major contributor to fine particle emissions even when particulate controls and FGD units are used.

Discussion

The fact that two fragmentation modes larger than 0.5 μm diameter, or three distinct modes in all, were observed using different particle-sampling and -sizing techniques (SMPS, APS, and two LPI models) suggests that the existence of three modes is not an artifact of the measurement technique. Furthermore, the EPA combustor data have sufficiently high resolution to show that the two fragmentation modes are so cleanly separated that there exist, in many cases, almost no particles with a diameter of 4–5 μm. This has profound implications on the pertinent mechanisms forming the fine fragmentation mode. A question could be posed as why 4–5 μm particles fail to form, rather than the converse question of why 1–2 μm particles do form?

Scanning electron microscopy (SEM) micrographs were obtained for a selection of particles on stages 3, 8, and 10 of the LPI for several coals burned in the UA combustor. These correspond to ash particles sizes contained in the ultracine, fine fragmentation (central), and coarse modes, respectively. The morphology of the ultracine particles (stage 3) depicted tiny smooth spheres and agglomerates consistent with a mechanism involving ash vaporization and subsequent aerosol nucleation and growth. Examination of micron-scale particles (stage 8) collected at postcombustion zone conditions for the Pittsburgh, Ohio, and Wyodak coals indicate that a significant number of the particles were rounded in nature. However, unlike the skeletal ash fragments and spheres observed in the large-diameter (coarse mode) fly ash samples (stage 10), many of these micron-scale particles were deformed oblates and showed other shape irregularities. While the surfaces and edges of these particles appeared softened and rounded, they had not entirely melted. They could, however, be the consequence of fragmentation mechanisms involving particle inflation,
cracking, and material shedding due to expansion of gases within the coal particles (cenosphere fracture) or through the formation and shedding of partially melted appendages or mineral inclusions during char combustion.

Re-examination of literature data shows that the central, fine fragmentation mode did occur, even though the resolution then available often led investigators not to draw attention to it. For example, re-examination of the field data of McElroy et al. [8] from a small 25-MW unit clearly suggests a fine fragmentation mode of ash particles at 3 μm diameter, even though discussion there centered on only one ultrafine/vaporization mode and one coarse/fragmentation mode. Helble and Sarofim [10] drew attention to the presence of a fine fragmentation mode in the ash PSDs obtained from their drop-tube furnace. They point out that shedding of ash from the particle surface was much more important than had been recognized previously. Fig. 4 shows the McElroy et al. [8] data on the lower panel, and the MIT data [10,23] on the upper panel. In all cases, there are two fragmentation modes (three modes in all, including the ultrafine mode). These literature data are consistent with other re-examined data from the literature [7], and so it is reasonable to conclude that the fine fragmentation mode is a real, practical phenomenon. The presence of a fine fragmentation mode is important for the broad range of coals tested as well as in full-scale units, especially in the light of current concern regarding fine PM emissions and control.

From the foregoing discussion, it is clear that the observation of more than one fragmentation mechanism is not new. Helble and Sarofim [10] attribute the fine fragmentation mode to ejection of molten ash particles from rapidly rotating char particles. The rapid rotation was due to jets of volatile organic compounds bursting from coal particles during devolatilization. This mechanism would produce two ash fragmentation modes, one resulting from coalescing ash within the parent particles, and the other from the fragmented molten ash material. Based on this model, one would expect the morphologies of all these ejected molten ash particles to be pure spheres. However, the SEM micrographs from the samples examined here indicate a combination of rounded oblates and spheres.

The Newcastle group [11] has shown the existence of char cenospheres from combustion of Australian bituminous coals, and suggest that smaller ash particles arise from char burnout of the thin shell. In order for two ash particle size modes to occur, some char particles must form cenospheres while others (the majority) must not. Kang et al. [13] suggest that this process depends on heating rate. Baxter [12] also supports this hypothesis, and has developed a theoretical framework to predict ash PSDs. However, for this mechanism to be solely controlling, one would expect a strong dependence on coal quality and the propensity to form hollow carbonaceous spheres. While this mechanism may be contributory for certain coals, the data presented here for 11 coals indicate little dependence on coal quality and the presence of three modes for melting and non-melting coals alike. There is also no evidence of bimodal inlet coal PSDs being a potential source for two fragmentation modes for the coals examined [24].

Rather, the SEM micrograph evidence indicates a mechanism (or mechanisms) involving secondary fragmentation of a portion of the friable ash, where temperatures are insufficient to achieve complete melting thereafter. Why the upper size of these fragmented particles is limited is less obvious. It may be related to a characteristic inclusion diameter, or there may be fundamental constraints on the shapes and sizes of friable appendages that form on the surfaces of burning char. The percolation approach adopted by Kang et al. [25] and others might well be able to yield the necessary insights into this process.

Conclusions

Ash formed from pulverized-coal combustion is generally defined by three modes. The smallest ultrafine mode is the result of well-established ash vaporization, nucleation, and growth mechanisms. The
largest coarse mode, also consistent with classical theories, arises from agglomeration of ash within the burning char particles. Finally, data presented here indicate a distinct, central, fine fragmentation mode that does not necessarily depend on coal composition, but comprises the major portion of primary fine particle mass emissions from pulverized-coal combustion. However, while these results are supported by prior literature, the data are not exclusively consistent with either of the two mechanisms previously published, and may be the consequence of multiple mechanisms or a single fragmentation process which constrains the particle size within a narrow range.

The results presented here show that three modes are in fact the rule, not the exception, and that fragmentation mechanisms or a single fragmentation process which does not necessarily depend on coal composition, but comprises the major portion of primary fine particle mass emissions from pulverized-coal combustion.

Acknowledgments/Disclaimer

Portions of this work were sponsored by: the Japanese New Energy and Technology Development Organization (NEDO) under an International Joint Research Grant; the U.S. Department of Energy under contract DE-AC22-95PC95101 to Physical Sciences Inc.; and the U.S. Environmental Protection Agency under purchase order 0C-R093NASA with J.O.L. Wendt and contract 68-C-99-201 with ARCADIS Geraghty & Miller Inc. The authors gratefully acknowledge the cooperation of the University of North Dakota Energy and Environmental Research Center who provided the SEM analysis facilities. The research described in this article has been reviewed by the Air Pollution Prevention and Control Division, U.S. EPA, and approved for publication. The contents of this article should not be construed to represent Agency policy nor does mention of trade names or commercial products constitute endorsement or recommendation for use.

REFERENCES